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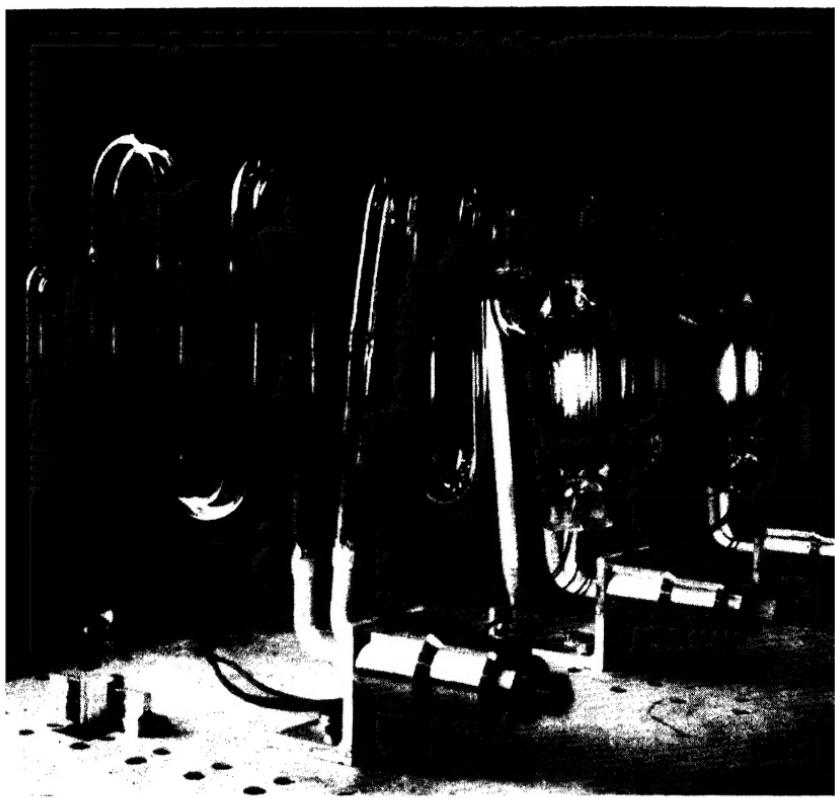
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HIGH VACUUM TECHNIQUE



Apparatus for obtaining Pressures of ca. 10^{-10} mm. of Mercury.
(Courtesy of Dr. D. Alpert and Westinghouse Research Laboratories, East Pittsburgh,
Pennsylvania, U.S.A.)

HIGH VACUUM TECHNIQUE

*Theory, Practice, Industrial Applications
and Properties of Materials*

BY
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THIRD EDITION

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PREFACE TO THE THIRD EDITION

A further increase in the size of this monograph has been undertaken to discuss the innovations in vacuum practice which have taken place since the second edition of 1945. Amongst the new data considered, the following topics are noteworthy : an extensive analysis of vacuum pumps available from both British and American manufacturers ; the gas ballast pump ; further discussion of the theory of the diffusion pump in so far as such theory is of value to the user rather than the designer ; details of large diffusion pumps and systems ; new pump fluids ; new materials ; recent methods of using rubber gaskets ; modern types of valves, including compressed air and magnetically operated patterns ; innovations in low-pressure measurement techniques, and in methods of producing and measuring ultra-high vacua ; methods of leak-hunting using vacuum gauges.

Little has been added, however, to the sections dealing with the applications of vacuum ; this is because new applications have been so numerous that it becomes exceedingly difficult to describe them properly without making the book inordinately large. Moreover, many of these applications now demand considerable knowledge outside the sphere of vacuum practice proper if they are to be fully comprehended.

The author again records his thanks to manufacturers of vacuum apparatus, in this country, in America and in Germany, who have kindly supplied so much valuable information.

J. YARWOOD.

PREFACE TO FIRST EDITION

The technique of high vacuum has been admirably described in various monographs and scientific articles by physicists who have been largely responsible for the rapid growth of a new industry, electronics. The present work does not intend to compete with the classics on the subject, but rather to introduce the latest developments in apparatus, to describe important industrial processes hitherto discussed only in scientific periodicals, and to bring together in one volume the diverse facts regarding the relevant properties and uses of materials encountered in all types of vacuum work. At the same time the standard technique is described to give logical sequence to the text so that the physics student can appreciate the theory and practice of the art. The author owes a large debt to the various standard works, tables of physical constants, and scientific periodicals which it is hoped is sufficiently acknowledged in references and the bibliography.

The assistance of Mr. and Mrs. R. Brockman, Mr. G. Thomas and Miss D. Walker in preparing the manuscript is gratefully acknowledged.

J. YARWOOD.

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NOTE

The unit of pressure used throughout this book is one millimetre of mercury, called 1 mm. Hg.

1 atmosphere = 760 mm. Hg.

1 micron (μ) = 10^{-3} mm. Hg.

1 microbar = 1 dyne per sq. cm. = 0.75×10^{-3} mm. Hg.

CHAPTER ONE

The Production of High Vacua

The Mean Free Path. The behaviour of gases at low pressures is, in many ways, entirely different from that at normal pressures. This is chiefly because of the greater distances separating the molecules of the gas making the possibility of the molecules colliding with one another much smaller. So there is defined a useful quantity in work on gases: the mean free path; that is the average distance a molecule travels at the prevailing pressure before it collides with another. If d is the diameter of a molecule, then if the two molecules approach within a distance of d between their centres we may say they have collided; this may be written by saying that the target area of a molecule is πd^2 . If there are N molecules per unit volume, then the number in a thickness t of cross-section S is NtS and the target area is πNd^2tS . When $\pi Nd^2tS = S$, the target formed by the molecule fills up the whole area S , so that no molecule can traverse a distance t without making a collision. The value of t which makes $\pi Nd^2tS = S$ is

$$t = \frac{1}{\pi N d^2} \quad . \quad . \quad . \quad . \quad (1)$$

where t is the mean free path.

Maxwell showed that, considering the distribution of velocities among the molecules, a more accurate result for the mean free path was

$$L = \frac{1}{\sqrt{2} N d^2 \pi} \quad . \quad . \quad . \quad . \quad (2)$$

where L is the mean free path.

Since L is inversely proportional to N , so it is inversely proportional to the pressure of the gas at constant temperature. For air, a useful result is that

$$L = 5 \times 10^{-3}/p \text{ cm.}$$

where p is the pressure in mm. Hg.

Thus, at 1 mm. Hg, $L = 5 \times 10^{-3}$ cm. and at 10^{-3} mm. Hg, $L = 5$ cm.

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For an electron passing through a gas

$$L_e = 4\sqrt{2}L$$

where L_e is the mean free path for the electron.

Vacuum Pumps. Pumps for the production of high vacua may be roughly divided into two classes : (a) those which pump air from a vessel at atmospheric pressure, these are usually mechanical pumps ; (b) those which need a fore-vacuum, or pumps which begin to operate below a certain limiting pressure.

Only those pumps which are extensively used are described below. Other types exist in many forms, but they are rarely employed.

Pumps classified under (a) are commonly called "backing" or fore pumps. There are five widely used types : (1) the water-jet pump, (2) the Gaede pattern, (3) the Cenco-Hyvac model, (4) the Kinney pump, and (5) the gas-ballast pump.

The speed of a pump is measured at a given pressure, and equals the volume of gas pumped from a vessel at that pressure in unit time, where the volume is measured at the pressure.

(1) The Water-jet Pump. Water supply from a fast-running tap is fed through thick-walled rubber tubing into the nozzle at A. This water-stream emerges at high velocity from the converging jet B. The jet is surrounded by a cone to prevent splashing and also to guide the water-stream down to waste at C. A side-tube at D is connected to the vessel to be evacuated. Molecules of air in the region of B are trapped by the high-speed jet and forced into the atmosphere ; these molecules are replaced by further ones from the vessel undergoing pumping, and so on. By this means a pressure as low as 7 mm. of mercury is attained,

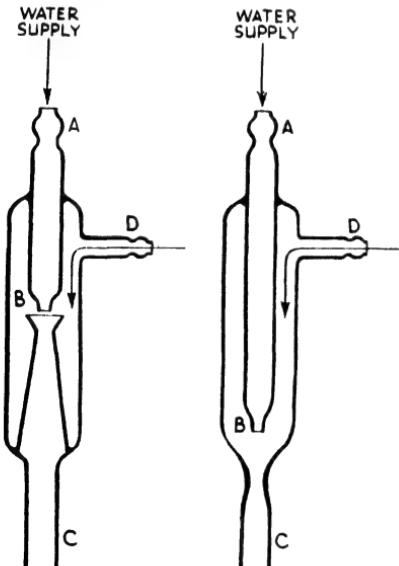


FIG. 1.—Water-jet Pumps.

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i.e. 5 mm. lower than the vapour pressure of water at room temperature.

Such pumps are made of glass or steel.¹ A drying agent should be provided (silica-gel or phosphorus pentoxide). This is conveniently placed in the tube connecting the pump to the vessel being evacuated. The pumping speed is of the order of 20 c.c. per second, at a pressure of 10 cm. of mercury, for a glass pump.

(2) **The Gaede Mechanical Rotary Pump.** A rotor consisting of a solid steel cylinder *P* rotates on its axis inside a cylindrical casting of bronze or steel *Q*. *A* and *B* are inlet and outlet ports respectively, i.e. the vessel being evacuated is joined to *A*, and the

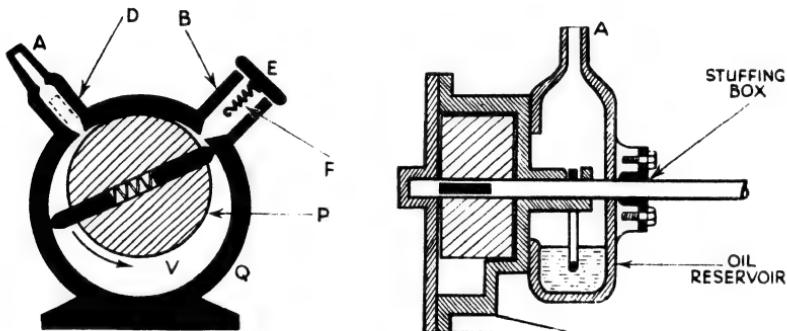


FIG. 2.—The Gaede Mechanical Rotary Pump.

air is sucked from it and expelled to the atmosphere at *B*. Over part of the area between *A* and *B* the inner and outer cylinders are in close contact. The inner cylinder rotates in a direction indicated by the arrow. Contact between the rotor and the cylinder is effected by two vanes inserted in diametrical slots in the rotor. These vanes are forced against the inside walls of the outer casing by means of springs.

The walls of the rotor and cylindrical case are worked to a high degree of precision. Sufficient oil is present to lubricate the moving parts and to act as a seal.

It will be readily seen from the diagram that, as the pump rotates,

¹ Steel and also plastic water-jet pumps are made by W. Edwards and Co., Ltd. They operate at a water pressure of 20 lb./sq. in., and incorporate a rubber non-return valve to prevent flooding of the vacuum system if the water pressure should fall.

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air originally occupying the volume V is compressed by the rotor and vane so that it is forced into the atmosphere at B . A fine wire sieve at D prevents foreign particles entering the pump and spoiling the precision-worked surfaces. A valve, consisting of a valve disc E , is held down on a valve seat by a spring at F in the outlet jet.

Pumping from atmospheric pressure, a pressure of the order of 10^{-2} mm. Hg is attained. With two pumps in series, i.e. one acting as a backing pump to the other, pressures as low as 10^{-4} mm. are obtainable (see Fig. 6).

An alternative form of Gaede pump is one immersed in a box,

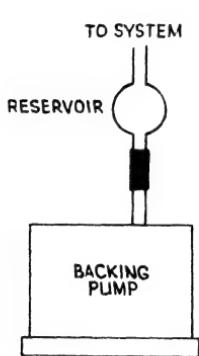


FIG. 3.

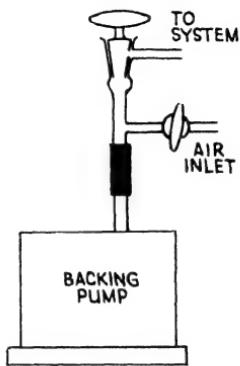


FIG. 4.

the whole mechanism being filled with oil which acts as a lubricant, and as an effective seal against small leaks (see Fig. 5).

When using oil-filled rotary pumps, care must be taken to ensure that, if the pump is left stationary over a long period, oil is not sucked back from the pump into any evacuated vessel connected to it. To prevent this, the pump is either fitted with a self-sealing oil-valve, or a reservoir is fitted above the pump as in Fig. 3, or the tap arrangement shown in Fig. 4 is adopted to allow the vessel to remain under vacuum whilst the oil-pump is at atmospheric pressure. Most modern pumps are fitted by the manufacturer with an oil-trap. They are often unreliable, however, especially when the pump has been much used, so it is advised that the precautions illustrated in Figs. 3 and 4 be adopted as a standard practice. The best precaution is undoubtedly to let the pump down to atmo-

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spheric pressure by the means shown in Fig. 4 because it is difficult to re-start a large pump under vacuum.

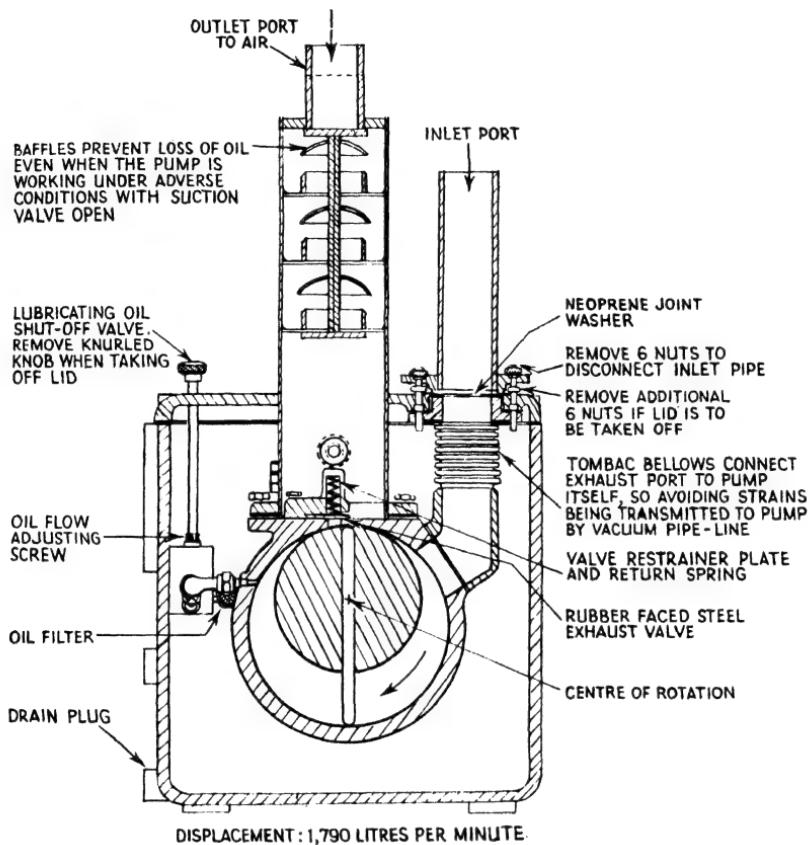


FIG. 5.—Large Rotary Vacuum Pump, Type SR5.

(Courtesy of Metropolitan-Vickers Electrical Co., Ltd.)

(3) The Cenco-Hyvac Rotary Oil Pump. This is a favourite pump in laboratories and lamp factories. Of American design, a rotor revolves inside an outer cylinder, but the rotor has a single spring-operated moving vane, and the inlet and outlet ports are close together.

Referring to Fig. 7, *A* is the inner, steel, fine-worked cylinder. This rotates eccentrically about the shaft *B* inside the steel cylindrical casing *C*. The vane is spring operated by the arm *D*. The

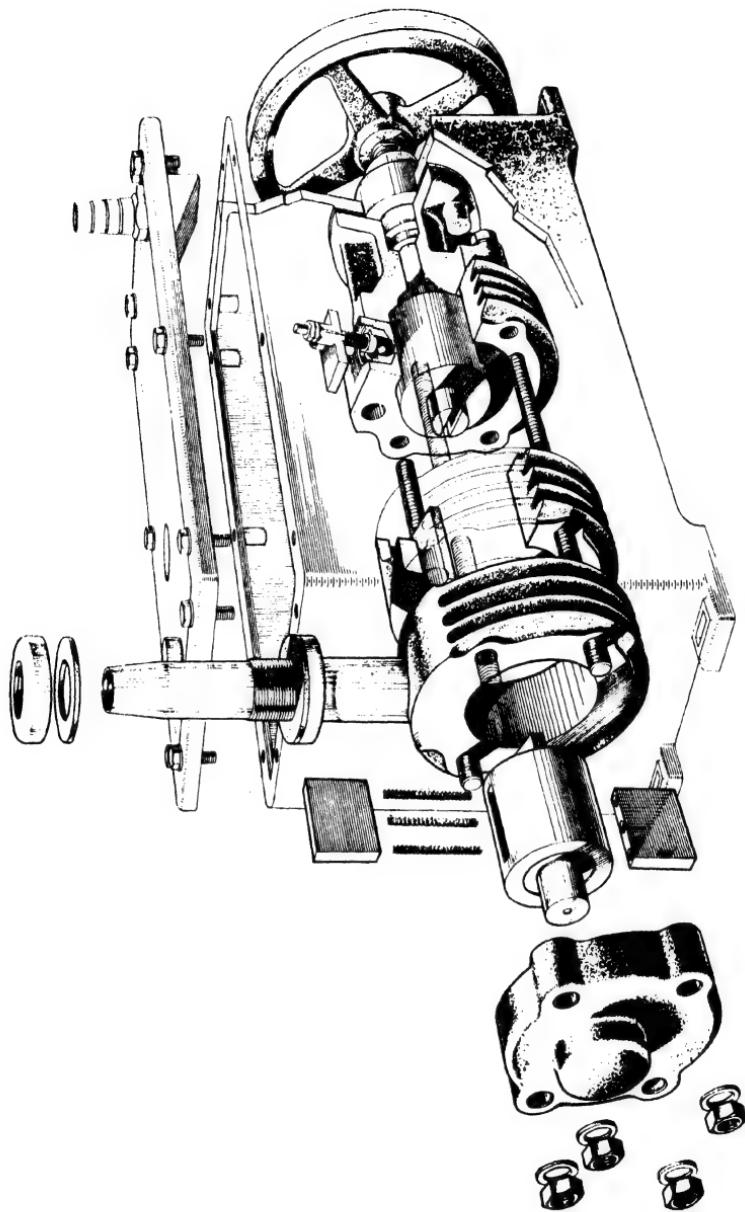


FIG. 6.—Construction of Two-stage Speedivac Pump.
(Courtesy of W. Edwards and Co., Ltd.)

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arrow on the rotor indicates the direction of movement. As in the case of the Gaede pump, it will be seen that air, in contact with the vessel being evacuated via port *E* and occupying volume *V*, is forced by the movement of the rotor and vane into a smaller volume near the outlet valve *F*, and so into the atmosphere. Many such revolutions quickly evacuate the vessel down to a pressure of 10^{-3} mm. Hg. The whole mechanism is immersed in oil in the box *G*.

The commercially made "Hyvac" consists of two such units mounted side by side on a common motor-driven shaft; the two pumps are in series.

Other commercial elaborations of the "Hyvac" are the "Megavac" and the "Hypervac". Their performances are illustrated in the composite graph in Fig. 41.

The same precautions are necessary to prevent suck back of oil as in the case of the Gaede pump.

(4) **The Kinney Pump.** A mechanical rotary pump introduced in America by the Kinney Manufacturing Co.¹ is illustrated in Fig. 8.

Instead of employing moving vanes, a tube *F* of rectangular cross-section, which is a sliding fit in an auxiliary small cylinder, connects the gas inlet port to the rotor, or plunger. This plunger is mounted eccentrically about the motor-driven, revolving axle *E*, and is in two parts: the inner drum *C* rotates about the axle *E*, but the cylindrical shell *D* is a sliding fit on *C*, and since it is rigidly attached to the inlet sliding tube *F*, will not rotate with *C*, but undergoes a "rocking" motion whereby the point *G*, where there is close contact between the plunger and stator, sweeps round the inner wall of the stator.

As the plunger moves in the direction of the arrow it rapidly creates extra space at *A* into which some of the gas is admitted through the inlet port; simultaneously, compression of the gas

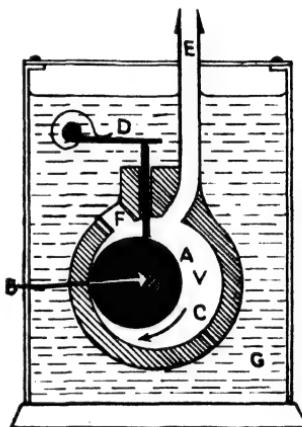


FIG. 7.—The Cenco-Hyvac
Rotary Pump.

¹ Also available in Britain from the General Engineering Co. (Radcliffe), Ltd., who kindly supplied catalogues on which the data given is based.

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previously trapped in volume *B* is taking place. When the plunger has almost reached its highest point it expels all air or gas and surplus sealing oil through the feather-type outlet valve and nozzle *H* into the oil separator tank, where the oil is retained and the air or gas is discharged into the atmosphere. With further movement of the plunger, the inlet port is completely closed, and the air or gas admitted to the full space *A* is trapped and then expelled by the next revolution, and so on.

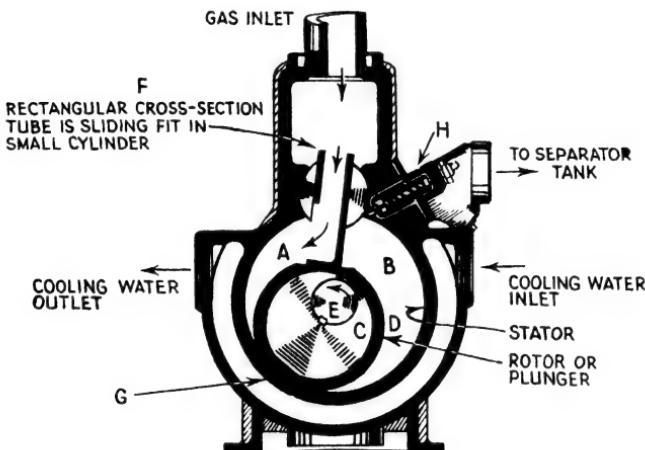


FIG. 8.—The Kinney Pump.

These pumps are available in large capacities (see page 50) and are water-cooled.

These mechanical oil-pumps are incapable of pumping condensable vapours such as water vapour, oil vapour, tap-grease vapour, etc. It is advisable to protect such pumps by a small boat of phosphorus pentoxide placed conveniently between the pump and the vessel. This prevents water-vapour spoiling the pressure, and also harming the precision-worked surfaces of the rotary pump.

(5) **The Gas Ballast Pump.** The object of this type of pump, first designed by Gaede¹ and marketed by Leybold's Nachfolger of Cologne, Germany, is to provide a mechanical rotary pump which

¹ See W. Gaede, *Eine Schrift aus dem Nachlass*, Leybold, 1951, and R. Jaeckel, *Kleinste Drucke, ihre Messung und Erzeugung*, J. Springer, 1950.

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does exhaust a system of water-vapour and other condensable vapours. In fact, it is possible, using a gas ballast pump, to begin with the inner walls of the vessel saturated with water and yet produce a vacuum of 10^{-1} mm. Hg in a matter of minutes, without using a drying agent like phosphorus pentoxide. Moreover, by the simple adjustment of an air-inlet device to the pump, the gas ballast mechanism can be used or not, at will, i.e. the pump either operates with gas ballast or as an ordinary rotary pump. Almost all the models of rotary pumps provided by Leybold's of Germany and by Gerätebau-Anstalt Balzers (G.A.B.) of Lichtenstein, have such gas ballast facilities. There is little doubt that, especially for larger pumps, this will also become the practice in England and America in the near future.¹

In the ordinary rotary pump, very high compression ratios of about 700 : 1 are commonly employed to ensure high pumping speed and low ultimate pressures. If the gases being pumped contain much vapour, condensation takes place even at low inlet pressures and the liquids formed mix with the oil. This oil then exerts a much higher vapour pressure than it does in the pure state so that the ultimate vacuum obtainable is poor. Moreover, the oil viscosity and its inertness to the working parts of the pump may be adversely affected. In chemical engineering practice, methods which are adopted to prevent this contamination of the oil include heating the pump to prevent excessive condensation, continuous renewal of the oil, and separation of the oil either by centrifuging or by circulating it around a separate oil-stripper unit. In dealing with excessive amounts of obnoxious vapours, such methods are likely to persist. For many purposes, however, the gas ballast pump is the best solution. In the gas ballast pump, the condensation is prevented by admitting air to the pump from the atmosphere at a suitable time in the operating cycle, so that the ratio by which the air-vapour mixture is compressed is only 6 : 1 or 10 : 1. Then, if the vapours enter the pump at less than 30° C. whereas the pump interior is at 60° to 80° C., the vapour does not condense at all, but is discharged with the admitted air through the outlet valve before condensation.

¹ Gas ballast pumps are now available from W. Edwards and Co. (London), Ltd., and from the National Research Corporation, Massachusetts, U.S.A. See page 49.

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To admit the gas ballast air to the pump at the correct time in the operating cycle, an air inlet valve, adjustable in size by a screw, is in the position shown in Figs. 9(a) and (b). Consider, then, the position *V* of the leading vane. Evacuation occurs over the part of one revolution from positions *A* to *B* because, when the vane is at *A*, the space between the pump cylinders below the vane is isolated from the inlet vacuum port and the space above the vane increases in volume from position *A* to *B*. When the leading vane arrives at *B*, however, then the rear vane *U* is at position *A* so that the space

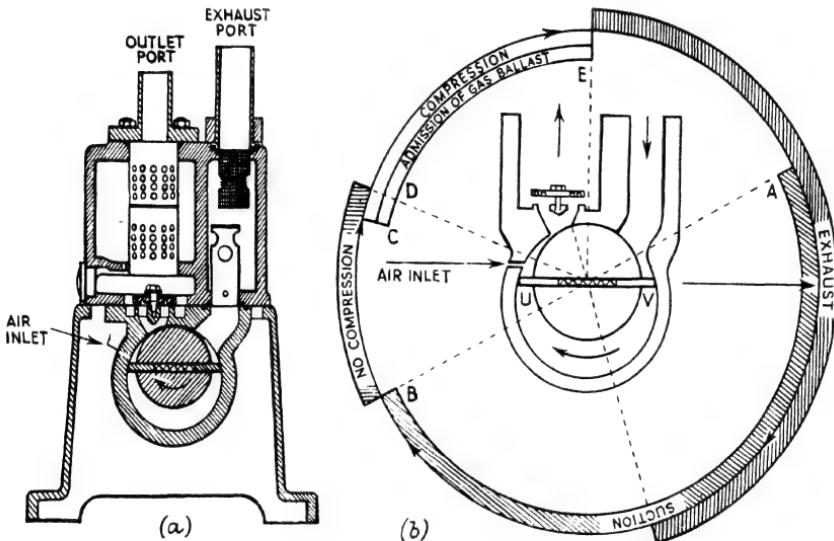


FIG. 9.—The Gas Ballast Pump.

above the leading vane *V* becomes isolated from the inlet port. During the part of the revolution from *B* to *D* there is no compression of the gas, therefore the isolated gas volume in the pump remains constant. So far, the operation is the same as in an ordinary rotary pump and, if the gas ballast were omitted, the compression to a high ratio of about 700 : 1 would occur in the part of the revolution from *D* to *E*. With gas ballast on, however, air from the atmosphere enters the pump at position *C*, just before compression begins to occur. From position *C* to position *E*, air is entering the pump via the gas ballast inlet at the same time as compression

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of the air-vapour mixture in the pump. The compression ratio is now only 6 : 1 or 10 : 1, depending on the type of gas ballast pump, when position *E* is reached. At *E* the pressure of the compressed gas exceeds 760 mm. Hg and the exhaust stroke begins. On further rotation of the leading vane, the gas-vapour mixture and the admitted gas ballast air are swept through the outlet valve.

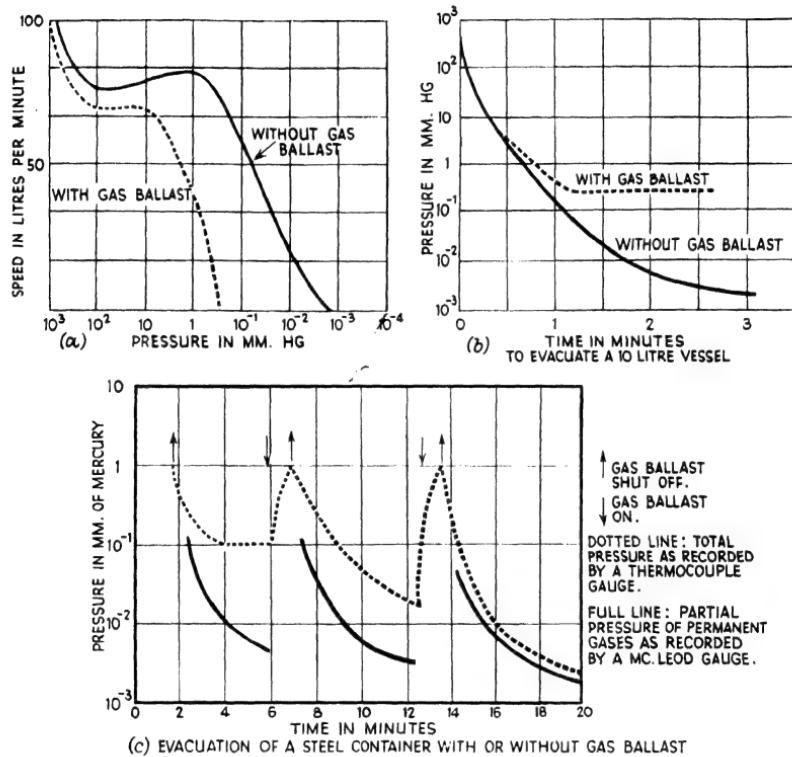


FIG. 10.—Performance of the Leybold S5 Gas Ballast Pump (Single-stage).

All condensable vapours are removed from the system by gas ballast pumps, but vapours which react chemically with the pump oil or the metal parts must be avoided.

As would be expected, the ultimate vacuum provided by the gas ballast pump is not at so low a pressure as that obtained with ordinary rotary pumps for dry air. A single-stage gas ballast pump

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provides an ultimate of 1 mm. Hg, whilst a two-stage model gives 5×10^{-2} mm. Hg. However, when the ultimate vacuum reached with the gas ballast in use is obtained, the condensable vapours have been largely removed and the gas ballast can easily be shut off by turning a valve. The ultimate pressures then attainable are the same as for ordinary rotary pumps, i.e. 5×10^{-3} mm. Hg for a single-stage and 10^{-4} mm. Hg for a two-stage pump.

The performance of the Leybold S5 gas ballast pump, (a) using gas ballast and (b) without gas ballast, is shown in the speed-pressure and pressure-time curves of Fig. 10. The advantage of a gas ballast pump in evacuating a steel container is shown in Fig. 10(c), taken from the catalogue of E. Leybold's Nachfolger, Cologne. Even though such a container is superficially dry, yet water vapour is released by the walls, and this water is only removed some time after using the gas ballast valve. Only then does the total pressure as recorded by a thermocouple gauge (*see page 99*) become equal to the partial pressure of the perfect gas, as measured by a McLeod gauge (*page 77*).

Pumps which require a Fore-vacuum. If it is required to remove considerable quantities of gas from a vessel at pressures less than 10^{-3} mm. Hg, then a rotary pump is inadequate because the speed is very small. Pumps which have high speeds at pressures less than 10^{-3} mm. Hg all require a rotary pump as well in series to provide a fore-vacuum of about 10^{-1} mm. Hg. There are two main types of such pump : (1) the molecular pump ; (2) the much more widely used diffusion-condensation pump which employs a working fluid which is either (a) mercury or (b) an oil of low vapour pressure.

The Gaede Molecular Pump.¹ The action of this pump relies on the dragging force exerted on molecules of gas by a rapidly moving surface adjacent to a stationary surface where both surfaces are worked to a high degree of precision.

Theory of Action. Referring to Fig. 11, a cylinder *A* rotates at high speed inside the outer cylinder *B*. The vessel is first evacuated to the fore-vacuum pressure, then when the cylinder of the molecular pump rotates, the molecules of gas in the vessel have a mean free path much longer than the spacing between the inner rotating cylinder and the outer casing of the molecular pump. The molecules therefore make many more collisions with the walls of the annular

¹ Gaede, *Phys. Zeit.*, 13, 864-70, 1912.

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gap than with each other. The result is that the gas is dragged from the vessel by the high-speed rotor and swept into the fore pump because the gas molecules take up the motion of the rotor

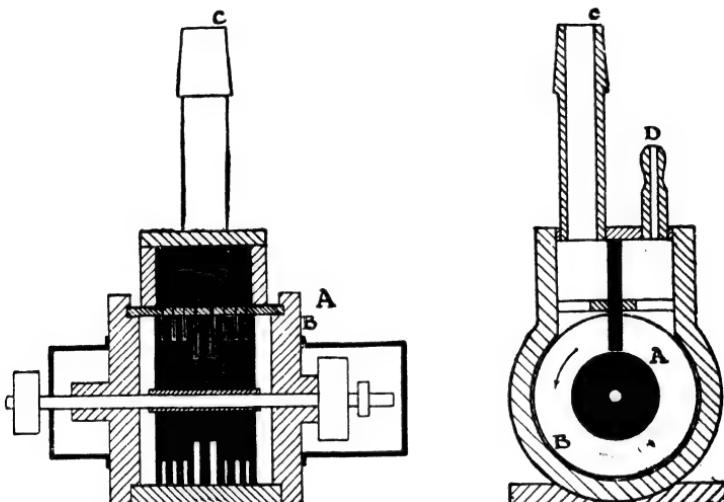


FIG. 11.—The Gaede Molecular Pump.

surface. There is a constant pressure difference between the openings *C* and *D*, *C* being directly connected to the vessel undergoing exhaustion, and *D* being the fore-vacuum connexion. This pressure difference is given by the equation

$$p_1 - p_2 = \frac{6l\omega\eta}{d^2} \quad . \quad . \quad . \quad . \quad (3)$$

where p_1 is the low-vacuum pressure, p_2 is the high-vacuum pressure, l is the length of the slot between high- and low-pressure connexions, η is the viscosity of the gas being pumped, ω is the angular velocity of rotation of the rotor and d is the depth of the slot. This equation follows from a consideration of the kinetic theory of gases.

As the fore-vacuum pressure is still further decreased, then the pressure-ratio between high and low pressures becomes independent of the fore-pressure and depends only on the speed of rotation ; it follows that reducing the fore-pressure below a certain minimum (10^{-2} mm. Hg) does not improve the performance. A pumping speed of 8 litres/sec. at 10^{-3} mm. Hg is attainable.

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The backing pressure is best about 10^{-2} mm. Hg. The rotating cylinder is furnished with about twelve slots into which ridges on the outer shell project. The clearance between the cylinders is approximately 0.1 mm. The slots are shallower at the outer section and deepen to about 0.5 cm. at the inner section.

The exhaustion is via port *C* and the backing pump is connected at *D*. The arrow indicates the clockwise rotation of the inner rotor. The rotation of the inner cylinder is usually effected by a high-speed induction motor. These pumps are fast and very convenient as no freezing trap is necessary as in the case of diffusion pumps; also they work under ideally clean conditions. They afford, however, quite a bit of mechanical trouble and need careful attention and maintenance.

The table below shows the effect of variations in fore-vacuum and rotor speed on the final vacuum produced. In this table *n* is the speed of the rotor in revolutions per minute, and p_1 and p_2 are the initial and final pressures in mm. of mercury.

<i>n</i>	p_1	p_2
10,000	2.0	$5 \cdot 10^{-7}$
10,000	10.0	$3 \cdot 10^{-5}$
8,000	20.0	$5 \cdot 10^{-3}$
4,000	10.0	$8 \cdot 10^{-2}$

Holweck's Pump. This is an improvement on the Gaede pattern. The inner cylinder is smoothly worked duralumin rotating inside a casing of bronze. The casing inner surface has a spiral groove cut into it. There are two grooves, one right-handed and the other left-handed, and they meet at the inlet port which is at the centre, see Fig. 12. The depth of the grooves increases from the ends, where it is about 0.5 mm., to about 5 mm. depth at the centre where the grooves join. The two outer ends of the grooves are connected through a channel cut in the casing to the backing pump tube. The gas being pumped is thus drawn by the rotation to each end of the cylinders. In Fig. 12, *A* is the inner rotating cylinder, *B* the outer casing, and *C* and *D* are the inlet and outlet ports respectively. The pump rotates at about 5000 revs. per minute and produces a vacuum of the order of 10^{-6} mm. Hg.

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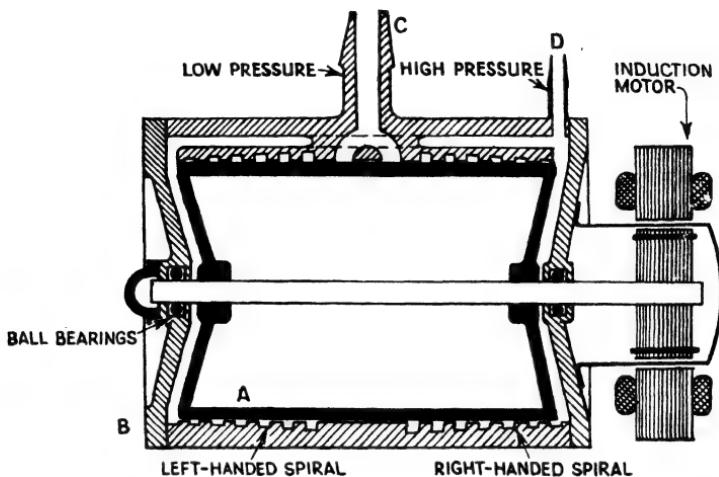


FIG. 12.—The Holweck Molecular Pump.

The Mercury Diffusion Pump. In this pump a stream of mercury vapour emerges from a jet. The vessel being exhausted is first pumped by the fore-pump through this diffusion pump before the mercury is heated. Then on boiling this mercury at the pressure produced (i.e. lower than 10^{-2} mm. Hg)

the high-velocity mercury vapour stream traps the molecules of gas which diffuse into it from the vessel, and guides them down to the fore-pump where the gas is passed to the atmosphere. The probability of a molecule not being conducted along by the stream has been calculated by kinetic theory to be about 1 in 10^{20} , so that the diffusion pump is very efficient.

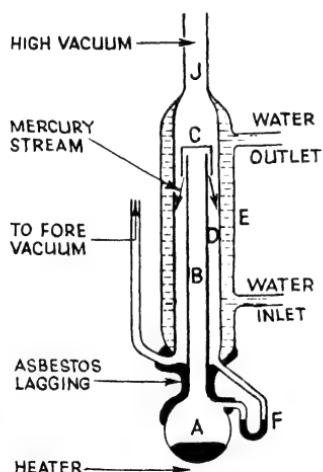


FIG. 13.—The Mercury Diffusion Pump.

This illustrates a diffusion pump called the "umbrella" type.

The mercury is boiled at backing pressure in the reservoir A

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and vaporizes into the tube *B*. At a suitable rate of heating, this stream impinges on the reflecting "umbrella"-shaped cone *C* and streams down the annular tube *D*. The pump is surrounded by a water-jacket *E* so that the downward stream of mercury finally condenses against the walls near the bottom of the tube *D* and returns to the reservoir by tube *F*. This tube *F* is U-shaped so that mercury collecting in it prevents direct contact between the backing pressure and the high pressure in the boiler *A*. The gas molecules in the space *J*, which is directly connected to the vessel being exhausted, diffuse into this mercury stream at *D* and are forced to the backing pump.

The diffusion pump is best called a condensation pump since the early Gaede diffusion pump did not include the condensing of the mercury vapour by the action of a water-jacket. Gaede's first diffusion pump needed a careful regulation of the mercury vapour temperature which, in the now universally used condensation pump, is not critical since the application of the condensation principle reduces any tendency of the mercury vapour to stream back into the vessel being exhausted. Although the temperature of the mercury vapour is not critical in the modern condensation pump (Langmuir¹) yet the rate of heating of the mercury determines the backing pressure needed for satisfactory operation, and must therefore be well regulated. Too slow a heating will require a low backing pressure, whilst too fast a heating will send the mercury vapour undesirably into upper parts of the pump. To apply the condensation principle satisfactorily, the end of the diffusion nozzle must be inside the water-jacket,² and the length of the pump great enough to prevent any appreciable quantity of gas diffusing back against the mercury vapour. The width of the slit from which the mercury diffuses determines approximately that the maximum pumping speed is attained at a pressure when the slit width equals the mean free path of the molecules of gas in the back-streaming vapour (see page 19).

Such pumps are made of glass, quartz or steel. Types in common use are depicted in Figs. 14, 15 and 16.

Fig. 14 is a pattern due to Volmer in which the direct downward stream is formed from mercury passing from the reservoir over the

¹ Langmuir, *Gen. Elect. Rev.*, **19**, 1060, 1916.

² Dushman, "High Vacuum", *G.E.C. Rev.*, 1922.

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tube T down to jet J , and is finally condensed by the Sprengel spiral at S . The tube T should be lagged with asbestos to increase the heating efficiency. This pump serves very well to act as an intermediary between the mechanical backing pump and an "umbrella" type pump, since the ultimate vacuum of a diffusion pump increases with a lower fore-pressure.

Fig. 15 illustrates a three-stage Gaede pump diffusion made of

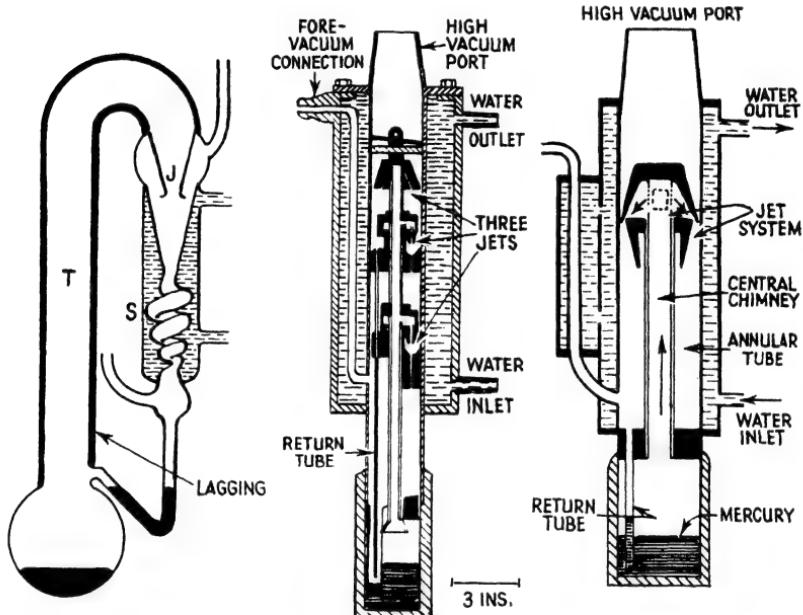


FIG. 14.—The Volmer Pump.

FIG. 15.—The Gaede Pump.

FIG. 16.—The Kaye Pump.

steel. Each stage backs the one above it, and the pump can thus be operated with a backing pressure as high as 20 mm. Hg and a final speed of 20 litres per second still maintained.

Fig. 16 illustrates a steel diffusion pump designed by Kaye.¹ It is a single-stage pump of wide bore to allow high-speed pumping without great restriction due to flow resistance of connecting tubes. Large-scale pumps built on a similar pattern have been made capable of a speed of 200 litres per second.

¹ Backhurst and Kaye, *Phil. Mag.*, **47**, 918, 1924; Kaye, *ibid.*, **1**, 349, 1926.

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Another model utilizes a precision steel-jet in a glass pump, so maintaining the cleanliness and convenience of glass when used for direct connexion to a glass vacuum tube, with the accuracy of a metal jet. See Fig. 17.

The relation between the theory of working and the design of diffusion-condensation pumps has been considered in several papers. It is not proposed here to deal with this subject extensively,¹ but to consider the theory only in so far as it affects the intelligent selection and use of pumps for various purposes.

Gaede² considers that, in all the various arrangements of jets used, the essential feature of the working is that mercury (or oil)

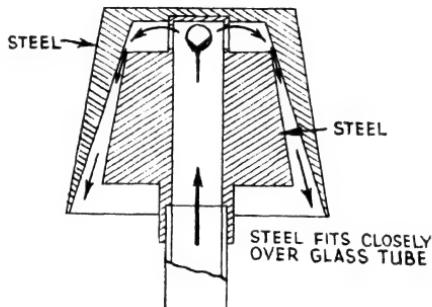


FIG. 17.

vapour in the boiler has a pressure depending on its temperature of the order of millimetres, and that it streams along a decreasing pressure gradient to the throat or slit, of width d , which has a form and dimensions depending on the pump geometry (ref. Fig. 18), where it is separated from the gas space at which gas molecules arrive from the vessel being exhausted. The width of this throat or slit is considered critical in so far as it decides whether the mercury vapour streaming through it will effectively give the gas molecules a decided component of velocity downwards towards the backing pump, so increasing the pumping speed, or whether mercury vapour will, by virtue of possessing too high a pressure, or due to incorrect slit width and disposition, tend to any extent to back-stream against the required pumping direction of gas molecules,

¹ For a full account, see S. Dushman, *The Scientific Foundations of Vacuum Technique*, Chapman and Hall Ltd., 1949.

² W. Gaede, *Z. tech. Phys.*, **4**, 337, 1923.

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and so reduce the pumping speed. There seems to be some confusion¹ existing as to the exact meaning of Gaede's statement

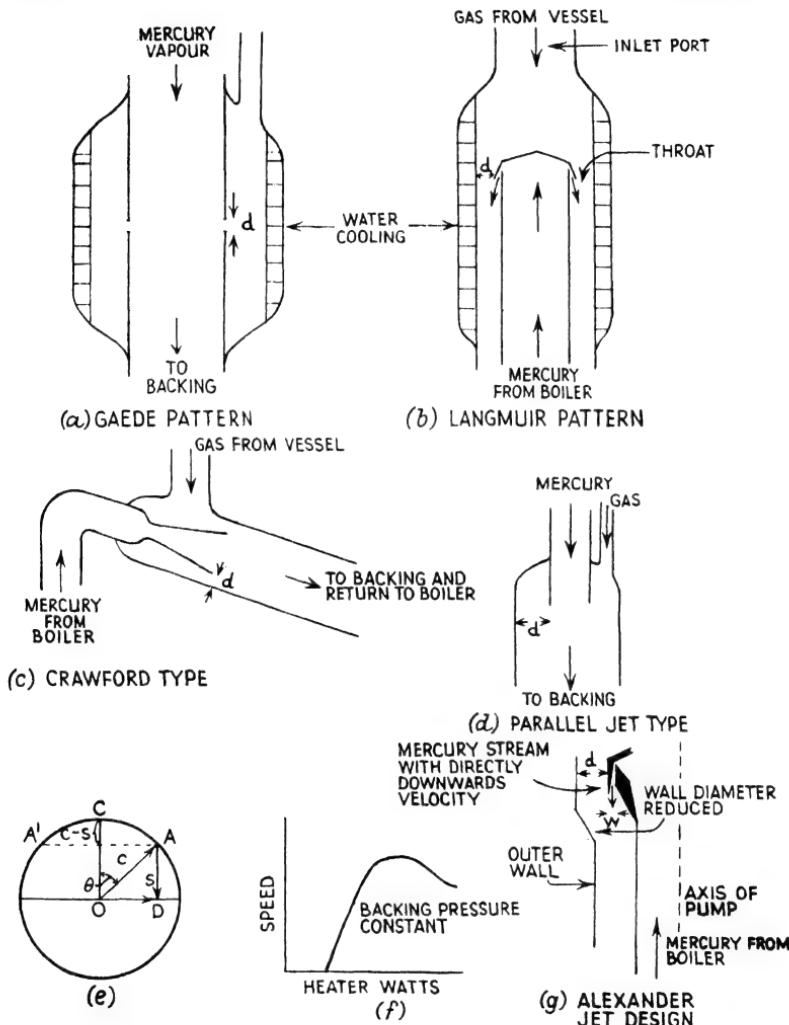


FIG. 18.—The Action of the Diffusion-Condensation Pump.

regarding the optimum slit width. In his earlier papers of 1915 he seems to imply that the mean free path of the vapour molecules

¹ D. Avery and R. Witty, *Proc. Phys. Soc.* **59**, 1016, 1947.

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in the stream shall be of the order of the width of the slit. This would demand slit widths of not more than 0·1 mm., limiting severely the pumping speed possible with a given inlet port diameter. Now many pumps have been built and successfully used with slit widths greater than this, yet without serious back-streaming of the vapour through the slit. The correct statement, given in his 1923 paper, is that the mean free path of the *molecules of gas in the back-streaming vapour* shall not be less than the slit width d . This allows much greater widths than 0·1 mm., since the mercury-vapour pressure would be about 1 mm., whereas the gas molecules in the backstreaming vapour would have a pressure of, at most, 10^{-2} mm., giving roughly an increase of possible throat width d of 100 times.

An account given by Alexander,¹ though not complete in that it assumes perfect gas laws hold, is instructive and useful in giving an insight into the nature of the requirements at the orifice. According to Maxwell,² the number of gas molecules N_{dc} moving with a velocity between c and $c + dc$ is

$$N_{dc} = \frac{4N}{\sqrt{\pi}} x^2 e^{-x^2} dx \quad (4)$$

where $x = c/\alpha$, α is the most probable velocity and N is the number of gas molecules per unit volume.

Since, in the absence of any directed stream of vapour, gas molecules are moving equally in all directions in space, so if all such molecular velocities between c and $c + dc$ are represented by vectors originating from one point, all the vectors will terminate on the surface of a sphere of radius c (Fig. 18e). If the figure represents such gas molecules in the gas space near the jet, then a downward directed velocity, s , due to the mercury-vapour stream, will be superimposed on this molecular motion. The particular vector drawn, OA , of length c , is such that, combined with the vector representing the vapour stream velocity, s , this molecule has a resultant vector OD in a horizontal plane. It is readily seen that all molecules within the upper segment ACA' of the sphere will have components of their resultant motion backwards, against the pumping direction. The number of these molecules must neces-

¹ P. Alexander, *Jour. Sc. Inst.*, **23**, 11, 1946.

² See, for example, J. K. Roberts, *Heat and Thermodynamics*, p. 63, Blackie and Son Ltd., 1943.

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sarily be a small fraction of the total number if adequate pumping is to be produced.

The number of backwards-moving gas molecules is those of which the velocity vector forms a smaller angle with s than θ . The vectors of such molecules end on the surface of a spherical cap of height $(c - s)$.

Let N_- equal the number of such backward-moving molecules. It is easily shown that

$$N_- = N_{dc} \frac{2c(c-s)}{4c^2} = N_{dc} \frac{c-s}{2c} \quad (5)$$

Put $s = a\alpha$, and $c = x\alpha$, where α = the most probable velocity, then

$$N_- = \frac{N_{dc}}{2} \left(1 - \frac{a}{x} \right) \quad (6)$$

and substituting for N_{dc} from equation (4), therefore,

$$N_- = \frac{2N}{\sqrt{\pi}} (x^2 - ax) e^{-x^2} dx \quad (7)$$

Evaluation of the integral of this expression between $x = a$ and $x = \infty$ gives the total number of molecules which spread backwards from the vapour stream. An approximate evaluation of this integral shows that, for every 1000 molecules present, the number moving backwards is 80 if $a = 1$, 17 if $a = 1.5$ and 2.3 if $a = 2$, corresponding to approximate pressures of the backwards spreading gas of 10^{-2} , 10^{-3} and 10^{-4} mm. Hg respectively.

Experiments performed by Alexander then showed that the streaming velocity of the vapour could be determined by finding the mass of mercury which passed through the jet in unit time, and employing calculations assuming the equation of state of an ideal gas, considered to be justified in view of the rarefied state of the vapour. The results of these experiments indicated that only when the jet width W (Fig. 18g) was large, was the streaming velocity less than 1.5 times the most probable molecular velocity, giving 17 in 1000 molecules back-diffusing against the pumping direction. A jet width W of 2.33 mm. could be permitted with an inlet port diameter of 9.5 cm. and a throat area of 143 sq. cm. with a heater input wattage of 4100, and yet a was still 1.5, so that only 17 in 1000 molecules diffuse backwards, whereas if the heater

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wattage was reduced to 2100, so reducing the mercury-stream velocity, yet a was still 1·4.

The demand for a high-velocity directed mercury-vapour stream to give efficient pumping was appreciated by Crawford¹ as early as 1917 (see Fig. 18c), who employed a divergent type of nozzle constructed, according to steam-engineering practice, so that the vapour-stream molecules were directed in the pumping direction with high velocities and nearly parallel directions. This type of jet, which will work well even without water-cooled walls, has been employed in many subsequent designs.

The theory of Alexander, indicating a demand for a high-velocity vapour stream, does not completely predict the effect of trying to increase this velocity by the use of excessive heater wattage at the boiler. An increasing number of collisions in the vapour stream will then increase the back streaming to an extent where the back-streaming vapour reduces the pumping speed, as well as cause vapour molecules to spread undesirably into the connecting tube to the vessel. As is shown in Figs. 18f and 43f, the speed of all types of pumps falls off as the heater wattage, and so mercury-vapour pressure with temperature, is increased above a certain value.

T. L. Ho² introduced the multi-nozzle diffusion pump (Fig. 19), maintaining that in pumps of the Langmuir and Crawford types, the condensing wall immediately below the orifice will scatter some of the gas molecules back into the chamber through that portion of the orifice which is adjacent to the condenser wall. In a multiple-jet construction, efficient use is claimed of all the vapour by using an enlarged nozzle chamber with sufficient space between the nozzles for the additional path of the gas. This, it is stated, gives rise to greater efficiency than would be expected on the basis of considering such an array as merely a set of small diffusion pumps. Thus a pump constructed with a single divergent nozzle, 6 mm. diameter at the entrance and 22 mm. diameter at the mouth, using mercury with 140 watts input to the heater, gave a pumping speed of 14·5 litres per second which was practically constant from 10^{-3} mm. to 10^{-5} mm. Hg, where the fore-pressure was 0·1 mm. Another pump, with the same overall nozzle size, but split into four parts, gave a pumping speed under similar conditions of 33 litres per second.

¹ W. Crawford, *Phys. Rev.*, **10**, 557, 1917.

² T. L. Ho, *Rev. Sc. Inst.*, **3**, 133, 1932.

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An important conception in the working of a diffusion pump has been clearly enunciated by Alexander (*ibid.*, 1946). In the vessel connected to the pump, the gas molecules will have velocities

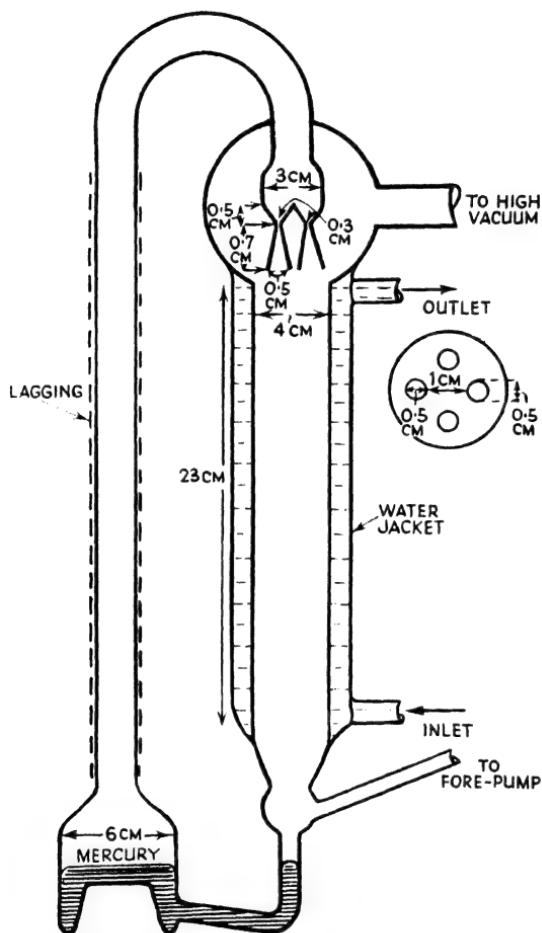


FIG. 19.—The Multi-nozzle Mercury Diffusion Pump (T. L. Ho).

corresponding to the temperature of the gas. At the jet, molecules will enter the mercury-vapour stream and receive increased velocities. Below the jet, the vapour gets less dense and its driving effect on the gas diminishes and finally ceases. The streaming

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velocity of the gas towards the backing outlet decreases likewise, so that its density increases. In short, the gas density is a minimum just below the jet, and a maximum near the backing outlet. There will therefore be a tendency for the gas to diffuse backwards from the high- to the low-density regions, against the pumping direction, and particularly along the pump inner walls where the driving effect downwards of the vapour is least. The mercury stream has to be dense enough to prevent this undesirable back-diffusion of the gas. An important criterion here will thus be the width of the pump throat, d , since above a certain width, the vapour stream along the wall will not be dense enough to "seal off" the compressed gas. These ideas were experimentally confirmed by obtaining lines of equal pressure, or isobars, in the space near and below the pump jet. To obtain a pump with a high throat width, and yet without such back-diffusion of the compressed gas along the walls, Alexander shaped the outer wall so that it was not parallel to the pumping direction (see Fig. 18g).

Alexander claims, by taking full advantage of the principles he has enunciated, to have made a pump with a speed of 1,400 litres/sec. in the pressure range 10^{-2} to 10^{-4} mm. Hg, with a maximum speed of 4 litres/sec. per sq. cm. of throat area.

Besides indicating the desirable features of a well-designed pump, this effect of "seal-off" indicates the necessity for a sufficiently low backing pressure. If the backing pressure is greater than the gas pressure built up by the mercury vapour forcing the gas towards the backing outlet, then gas from the backing side will diffuse backwards against the pumping direction of the vapour, particularly near the walls of the pump, and cause a serious reduction of pump speed due to destruction of the necessary pressure-sealing effect. If the backing pressure is below the critical value, then the diffusion pump speed is independent of the backing pressure. To obtain diffusion pumps which will operate with high backing pressures, a multi-stage jet system is therefore necessary, as in Figs. 15 and 31, or as in the specially manufactured fused silica, mercury diffusion pump of The Thermal Syndicate Ltd.,¹ which is designed to operate with a backing pressure adequately obtained by a water-jet pump, and so give a combination capable of producing a pressure of 10^{-2} mm. Hg, suitable for backing a single-stage diffusion pump.

¹ Ref. *Jour. Sc. Inst.*, 25, 245, 1948.

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Freezing Traps. To prevent any of the mercury vapour from entering the vessel undergoing pumping, and perhaps contaminating any structure therein, and also because mercury vapour has a pressure of 1.3×10^{-3} mm. Hg at 20° C., a freezing trap has to be inserted between the diffusion pump and such a vessel. Two common traps are shown in Figs. 20 and 21.

The freezing compound is either solid carbon dioxide mixed with ether (temperature -78° C.) or liquid air (-187° C.). The latter is preferable, since it is found that if the level of carbon dioxide in the trap falls during pumping, then the vapour pressure of any water

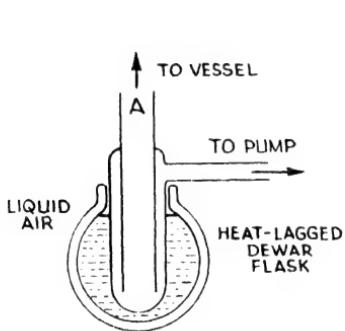


FIG. 20.—Freezing Trap.

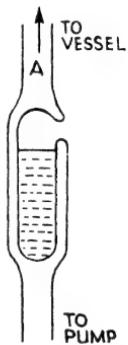


FIG. 21.—Freezing Trap.

condensing on the upper part of the trap in the region *A* in Figs. 20 and 21 is about 5×10^{-4} mm. Hg, which is a much greater order of pressure than the mercury diffusion pump is capable of producing.

It is emphasized that the diffusion pumps cope satisfactorily with condensable vapours which the backing pumps fail to withdraw from the system. Thus, keeping the freezing trap adequately clean is necessary to maintain efficiency.

It might be stressed at this point that a high standard of cleanliness is demanded in every type of vacuum work, since a minute speck of foreign material is, under low-pressure conditions, capable of producing several thousand times its volume of vapour.

The freezing trap is a handicap because it must introduce some opposition to the flow of gas from the vessel to the pump, and so lower the speed of pumping. This effect becomes very noticeable at low pressure and high pumping speeds. Attempts at a compromise between freezing efficiency and bore size partially solve the

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problem—thus the ratio of the diameters of the inner to outer tubes of the trap of Fig. 20 is best 0·6—but the oil diffusion pump, now to be described, is, for many purposes, the best solution.

The Oil Diffusion Pump. Both Hickman¹ and Burch² described the preparation of oils for use in diffusion pumps in place of mercury. These oils have a low vapour pressure compared with mercury, and they are comparatively inert chemically, so that in many types of vacuum operation the use of a freezing trap is avoided, and the pumping speed is unrestricted.

It must be realized, however, that these oils are ruinous to the activation of thermionic³ and photocathodes unless suitable precautions are adopted. To ensure that the oil does not enter the system in carrying out such operations, it is essential to employ some form of baffle cooled by waterflow, or preferably kept at 10° C. below room temperature, between the pump and the chamber (see page 30). Thus, a pump filled with Apiezon B oil and provided with a cold sink and baffle between the pump and apparatus will provide an unsaturated atmosphere of Apiezon B oil vapour in the vessel being evacuated, at a pressure necessarily lower than the saturation vapour pressure of the oil at room temperature, but further than this, it will then not be possible for the oil to condense as liquid drops in the vessel.⁴ Again, only oils of the lowest vapour pressure should be used, e.g. Apiezon B, Octoil S or D.C. Silicone 703. The Hickman self-fractionating column pump (Fig. 28) is useful to keep the oil free from decomposition products. On the other hand, for many industrial processes, e.g. metal deposition *in vacuo*, the electron microscope, and for the evacuation of large metal chambers generally, oil pumps with simple baffles are often satisfactory.

The molecular weights of the oils used are much greater than those of mercury, and the molecular volumes are greater by about fifteen times. For these reasons it should, according to simple

¹ K. C. D. Hickman, *Rev. Sc. Inst.*, **1**, 140, 1930, and *Jour. Frank. Inst.*, **221**, 215, 1936.

² C. R. Burch, *Proc. Roy. Soc.*, **123**, 171, 1929.

³ This does not apply when the cathode is activated after the tube is sealed off from the pump, as in the mass production of radio receiver valves. Again, especially in electron tube manufacture, the glass vessel is hot during the pumping time and precautions concerning back-streaming are not so necessary.

⁴ See Central Scientific Co. Bulletin 10A.

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theory, be possible to obtain greater speeds with oils than with mercury for diffusion pumps of corresponding sizes. This is not noticeably so in practice, indeed, in the case of some manufacturers' products the mercury pumps are slightly faster than the oil pumps of the same diameter. This effect is possibly due to Gaede's absorptivity factor, which takes into account the ability of the vapour stream to entrain and remove all the gas molecules which reach it, and also due to the fact that mercury with its greater thermal conductivity and higher boiling point can be more readily kept hot, and so have greater molecular speeds at the jets.

It is not the best practice to use a diffusion pump designed to be employed with mercury, and substitute an oil instead. This is because (1) the gap at the diffusion jet may be too small so that it becomes bridged by an oil film, (2) the cross-section areas of the jet and the tube supplying vapour to the jet may be inadequate to allow a sufficient supply of vapour ; also these parts of the pump may be at too low a temperature, causing the vapour to condense too soon.

Oils which have been satisfactorily employed in diffusion pumps are :

Oil	Specific gravity at 25° C.	Ultimate vacuum obtainable in mm. Hg	Boiling point at 10 ⁻² mm. Hg in °C.
Amoil S ¹ . . .	0·9251	2 × 10 ⁻⁶	111
" Apiezon " A ³ . .	0·8735 at 15° C.	10 ⁻⁵	111
" Apiezon " B ³ . .	0·871 at 15° C.	10 ⁻⁷	125
" Apiezon " G ³ . .		10 ⁻⁵	
Butyl phthalate ¹ . .	1·0465	4 × 10 ⁻⁵	80
Butyl sebacate ¹ . .	0·933	2 × 10 ⁻⁵	90
Myvane 20 . . .	0·853	10 ⁻⁶	
Narcoil 10 ⁵ . . .	1·54	10 ⁻⁵	
Octoil ¹	0·9796	2 × 10 ⁻⁷	122
Octoil S ¹	0·9103	5 × 10 ⁻⁸	143
Silicone D.C. 702 ² .	1·07	10 ⁻⁷	160
Silicone D.C. 703 ² .	1·09	5 × 10 ⁻⁸	200

The silicone fluids^{2, 4} developed by Dow Corning are semi-organic polymers which, in addition to having high molecular

¹ Data by courtesy of Distillation Products Inc., New York, U.S.A.

² Courtesy of Dow Corning Corp., Michigan, U.S.A.

³ Courtesy of Apiezon Products Ltd., Great St. Helens, England.

⁴ G. Brown, *Rev. Sc. Inst.*, **16**, 316, 1945.

⁵ Courtesy of Vacuum Industrial Applications Ltd., Wishaw, Scotland.

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weights and low vapour pressures, are exceptionally resistant to oxidation at high temperatures. As a result, heating of these oils at atmospheric pressure does not seriously impair their efficiencies as pump fluids. Unlike the other organic fluids listed above, D.C. 702 and D.C. 703 do not decompose even on exposure to air at operating temperatures. Such drastic treatment of the truly organic fluids results in the formation of tars and corrosive vapours in the diffusion pump which block up jets, and stick to the working surfaces, so reducing noticeably the working efficiency. Using silicones, on the other hand, any unfortunate or inevitable exposure of the hot pump fluid to the atmosphere does not cause serious decomposition or chemical change, so that even after twenty minutes' exposure to atmospheric pressure at the operating temperature, they will subsequently produce a high vacuum almost as rapidly as before. They also show little evidence of chemical change on being heated in contact with aluminium, copper, steel or glass. The pumping speed characteristics and fore-pressure requirements of these oils are otherwise similar to those for the usual organic liquids, except that speeds are somewhat lower for the same heater wattage because of their higher boiling points. They are strongly recommended for demountable and large-scale industrial gears where the exposure of the hot pump oil to pressures above 10^{-1} mm. Hg is necessary to speed up the operating cycle of the plant.¹ However, though the author has not conducted extensive trials, it appears from experimental work that silicone oils should not be deliberately exposed repeatedly to air at atmospheric pressure when hot if their high performance is to be maintained.

The silicone oils are very valuable additions to the range of working fluids for diffusion pumps. However, it is important to realize that they do give difficulties due to back-streaming. Several cases have been recorded where, in experimental work on electron tubes, mass spectrometers, ion accelerators, etc., back-streaming of small quantities of silicone oils into the system have become deposited on electrodes and, on heating, electron or ion bombardment, have resulted in conducting surfaces becoming coated with insulating layers of silica and compounds containing silica.

¹ See D. Latham, B. D. Power and N. T. M. Dennis, "An Assessment of Some Working Fluids of Diffusion Pumps", *Vacuum*, 2, 33, 1952.

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H. W. Edwards¹ describes a metal-type oil-diffusion pump in which the chief object of the design is to provide stream-lines in the higher pressure parts of the pump, and smooth guiding surfaces which offer a minimum impedance to the flow of gases in those

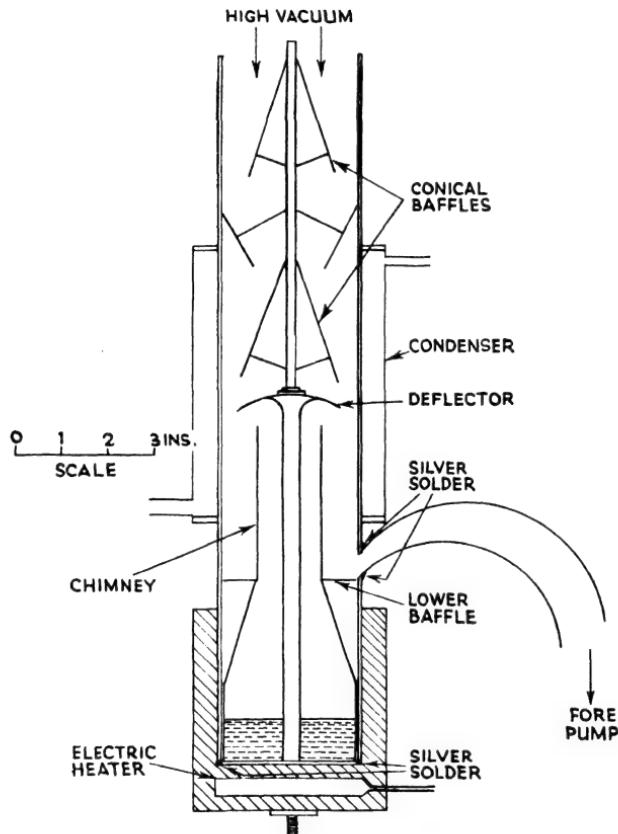


FIG. 22.—A Metal Oil Diffusion Pump (H. W. Edwards).

parts of the pump at the lowest pressures. The arrangement is shown in Fig. 22.

The internal construction is of copper, the most suitable metal since it is unattacked by the oil,² and has good heat conductivity.

¹ H. W. Edwards, *Rev. Sc. Inst.*, **6**, 145, 1935.

² The copper is preferably nickel or chromium plated because copper, brass, zinc and tin induce chemical reactions catalytically with some oils, in particular, butyl phthalate and Narcoil.

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It is noteworthy that silver solder joints at the base of the pump, and where external tubes are connected to the body of the pump, enable this construction to be made with comparatively limited workshop facilities. A clearance of about 1 mm. between the lower part of the pump "chimney" and the pump body permits condensed oil to return to the reservoir. A lower baffle about 0.5 mm. thick is mounted with a very small clearance between its circumference and the pump wall so that oil collects there to act as a seal to prevent oil vapour rising outside the chimney. A clearance of 7.5 mm. between the outer edge of the deflector and the pump wall exists, which can be increased to some extent if a very low pressure is required, though a lower backing pressure then becomes necessary. Between the upper edge of the chimney and the under surface of the deflector there is a clearance of about 6 mm. in the vertical direction. Some small wires spaced round the edge of this deflector prevents oil drops collecting, since drops will drain off along such wires.

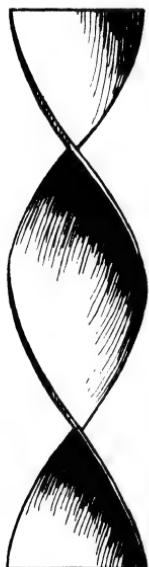


FIG. 23.—
A Spiral Baffle.

A conical baffle at the throat of the pump prevents oil vapour getting into the system undergoing evacuation, and offers little impedance to the downward flow of gases. A spiral baffle was also used (Fig. 23). This pump is stated to have a pressure limit at 10^{-7} mm. Hg, and a speed of 50 litres per second at 10^{-3} mm. Since the pump is a single-stage one, a low fore-pressure should be used, not higher than 5×10^{-2} mm. Hg.

R. M. Zabel¹ built a multi-nozzle pump of the Ho type (see page 22), using oil. A short wide tube from the boiler to the nozzles ensured an ample vapour stream without requiring that the boiler be heated excessively, which would decompose the oil. A second advantage of Zabel's arrangement is that the hot tube through the condensation chamber is avoided by adopting a horizontal construction. Fig. 24 gives the details and dimensions of this pump if four nozzles are used. A greater number of nozzles can be successfully employed in a construction of wider tube diameter, and so greater pumping speed.

¹ R. M. Zabel, *Rev. Sc. Inst.*, 6, 54, 1935.

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J. E. Henderson¹ describes a two-stage oil diffusion pump which will operate with higher backing pressures. The chief point to be noted in this design, illustrated in Fig. 25, is that the first stage jet is arranged to slope continuously downward so that droplets of oil do not collect, which allows the small clearances necessary for the poor backing vacuum. The clearance between the walls and the nozzle is adjustable by means of the screw at the base of the pump, so that an optimum performance can be obtained depending on

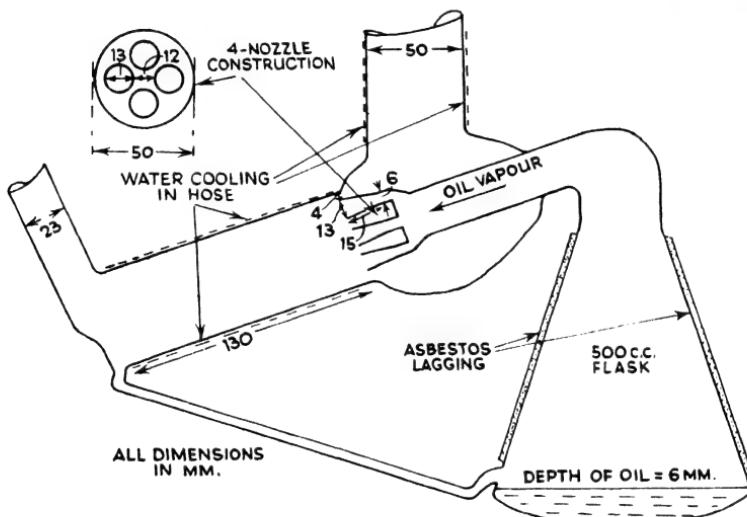


FIG. 24.—A Multi-nozzle Oil Diffusion Pump (R. M. Zabel).

the backing pressure. Other considerations of this design are indicated by the captions on the diagram.

J. A. Bearden² made a pump of the Zabel type with a single-stage straight nozzle. The most noteworthy feature of this pump was the re-entrant, concave type of boiler enabling atmospheric pressure effects to be rendered harmless, and an immersion heater to be used with the minimum of oil (*see* Fig. 26).

A study of the speed of divergent nozzle pumps has been made by Copley, Simpson, Tenney and Phipps.³ The relation between

¹ J. E. Henderson, *ibid.*, 66, 1935.

² J. A. Bearden, *Rev. Sc. Inst.*, 6, 276, 1935.

³ Copley, Simpson, Tenney and Phipps, *ibid.*, 265, 1935.

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pumping speed and the annular area between the end of the nozzle and the condensing walls of the pump was investigated. The speed was correctly shown to increase with the annular area, even for the largest areas studied, which is in contrast to the earlier results of Estermann and Byck¹ who worked with the "umbrella" type of

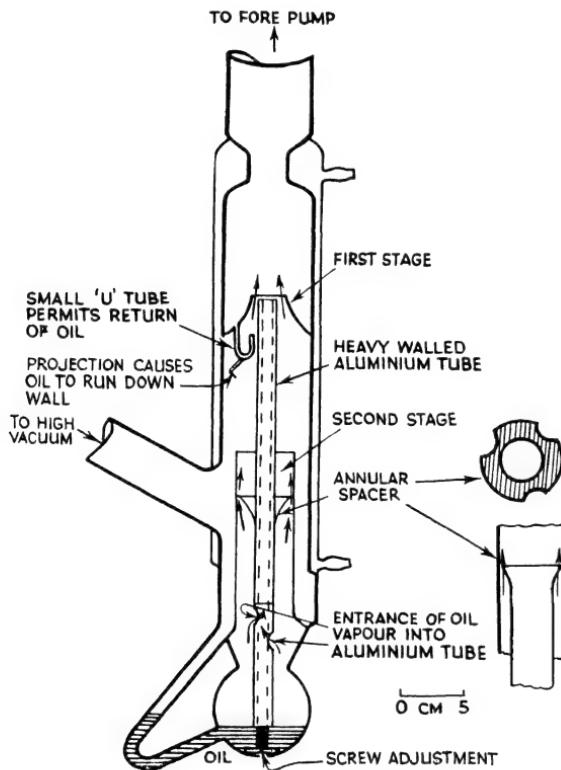


FIG. 25.—A Two-stage Oil Diffusion Pump (J. E. Henderson).

construction, and showed that the speed was a maximum, for a given pump, with an annular area of 7.4 sq. cm. corresponding to an annular width of 0.55 cm., and fell off rapidly for the larger areas.

A speed factor for pumps, given as the ratio of the speed measured at the inlet to the jet chamber to the ideal speed as calculated from

¹ Estermann and Byck, *Rev. Sc. Inst.*, 3, 482, 1932.

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the kinetic theory of gases,¹ was shown to be a maximum for divergent nozzle pumps when the annular width was 0·76 cm., and the annular area was 8·0 sq. cm.

The long divergent type of nozzle was considered to be superior to the short divergent type, whether single or multiple, with regard to this speed factor. The nozzle construction adopted by Copley and his co-workers is shown in Fig. 27.

There is at present some disagreement amongst designers as to which is the most satisfactory nozzle construction.

Some more extensive work of a practical character based on a sound theoretical foundation is required to settle finally some points, but

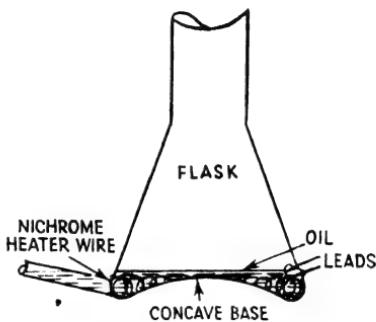


FIG. 26.—A Pump Boiler of Novel Construction (J. A. Bearden).

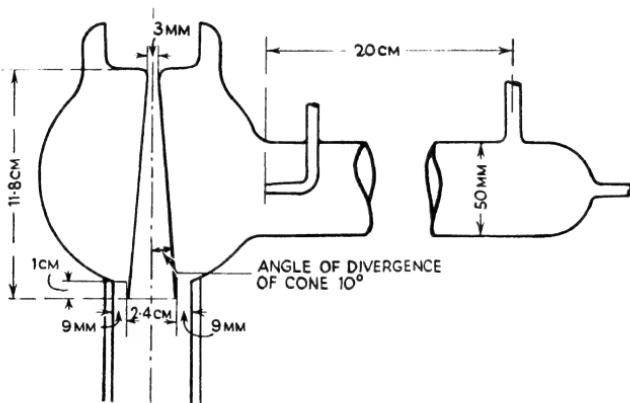


FIG. 27.—The Long Divergent Type of Nozzle (Copley).

on the whole, the long divergent nozzles, and cylindrical types, seem the most satisfactory arrangements for oil diffusion pumps.

¹ The maximum theoretical speed in litres per second is 11·7 times the area of the throat in square centimetres, i.e. the annular area between the jet system and the walls; 11·7 litres/sec. is the rate at which air at pressure P will flow through a hole of 1 sq. cm. area into a region where the pressure is p . This rate is independent of the pressure P provided that $P \gg p$ and that the volume concerned is measured at the pressure P . The speed factor is known as the Ho coefficient. Maximum practical values of this coefficient are 0·4 approx.

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A pump designed by Hickman¹ uses a two-stage arrangement, one stage of the long divergent nozzle type, the other a cylindrical nozzle. This pump (Fig. 28) is combined with a fractional distillation apparatus which maintains the oil uncontaminated and at very low vapour pressure. Immersion heaters are used. The oil will necessarily have components and impurities with different vapour pressures and so somewhat different boiling points. The

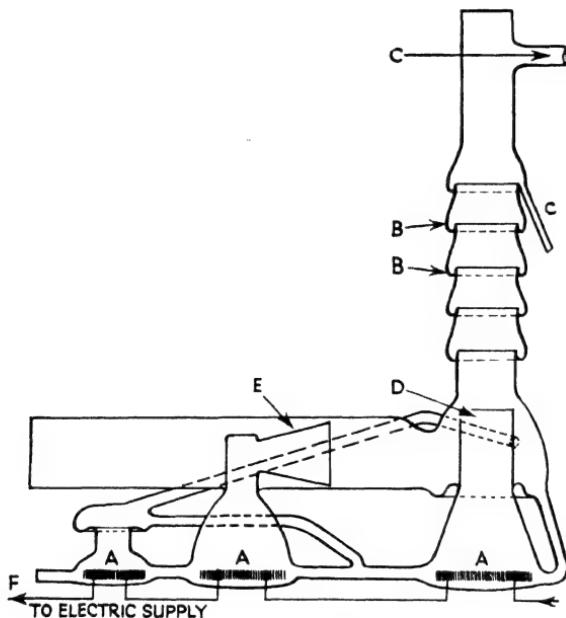


FIG. 28.—The Fractionating Oil Diffusion Pump (Hickman).

most volatile constituents are distilled into the alembics *B*, those of medium volatility form the vapour streams at the jets *D* and *E*, whilst the unvolatile components and impurities go to the small boiler at the end of the pump where the dark-coloured, non-volatile constituent collects and the more active components are distilled and returned to the other two boilers. *D* is the second-stage jet, *E* the first-stage, high-vacuum jet. Thus the fore-pump is connected at *C* and the low pressure pertains at *E*.

The wiring to the bare wire heaters is via *F*. The horizontal type

¹ K. C. D. Hickman, *Jour. Appld. Physics*, May, 1940.

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of construction is superior to the vertical pattern in this case since the jet diameter is independent of the boiler area, and the heat input to the separate boilers can be precisely regulated.

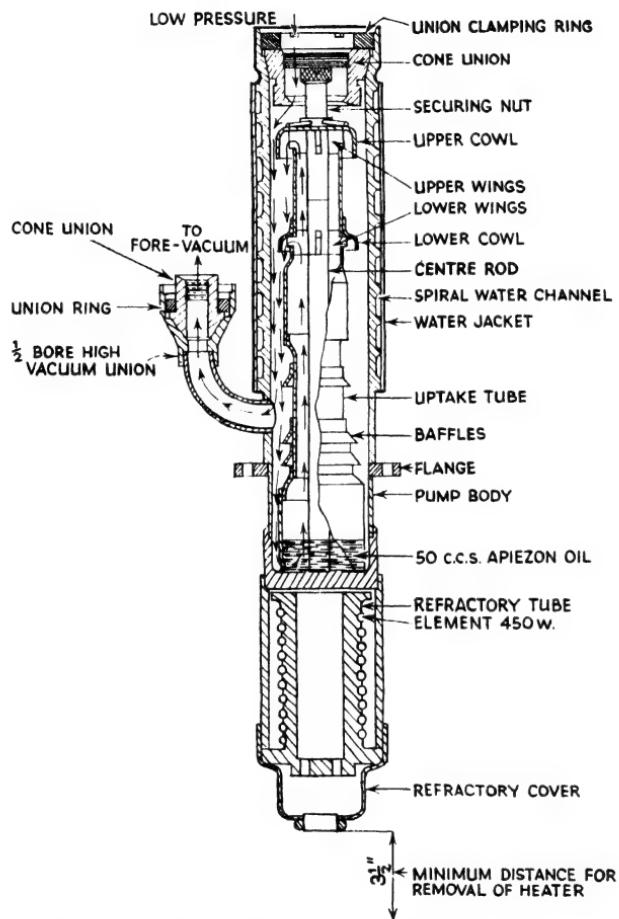


FIG. 29.—The Metro-Vick 02 Oil Diffusion Pump.

(Courtesy of the Metropolitan-Vickers Electrical Co., Ltd.)

A two-stage oil diffusion pump manufactured by the Metropolitan-Vickers Electrical Company is illustrated in Fig. 29. The outside main tube of the pump is of steel, but the jet system is made of copper because of the advantage associated with its high thermal

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conductivity. It is intended to be used with the Apiezon oils. The 02 type is capable of a maximum speed of 7 litres per second at a pressure of 10^{-3} mm. Hg, but an 03B pump is available which, backed by the 02 in a series combination, is capable of a speed of 30 litres per second, and a final pressure of 10^{-6} mm., using the B type oil.

Oil diffusion pumps with speeds as great as 5000 litres/sec. at 10^{-3} mm. Hg are commercially available, whilst pumps with inlet diameters of 24" and speeds of 12,000 litres/sec. have been built.

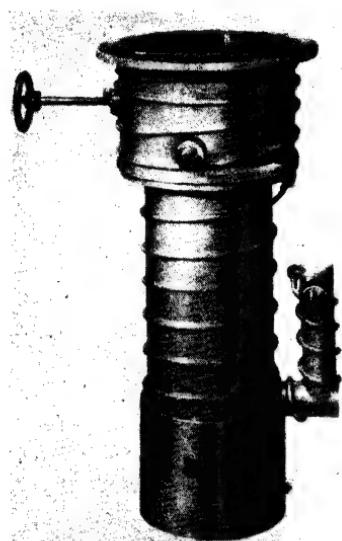


FIG. 30.—A Large Oil Diffusion Pump, 903A (W. Edwards and Co., Ltd.).

and the mechanical pump. The lower jet has to operate at a higher pressure than the top jet, does not require to have such high speed (ref. page 39) but must not allow the vapour passing through it to expand so much that the relatively high backing pressure produced at speed by the mechanical pump is prohibited. It therefore has a smaller area, there being less clearance between its edge and the inner wall of the casing.

A type of diffusion-ejector pump, known as a booster, is designed to act as an intermediary between the mechanical pump and a large oil diffusion pump. Such boosters are usually single-jet pumps

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using an oil such as butyl phthalate, which will not decompose readily at the boiler temperature required to operate a pump with a high speed at some 10^{-1} mm. Hg, and will operate with a backing pressure as high as 1 mm. Hg. A booster pump built as an integral part into the tube leading from a large two-stage diffusion pump to the mechanical backing pump is often used in commercial practice. Fig. 32a shows schematically an oil-ejector booster pump built by Distillation Products Inc.,¹ the type KB-300. This has a speed of 350 litres per sec. at a pressure of 2×10^{-1} mm. Hg (see page 52).

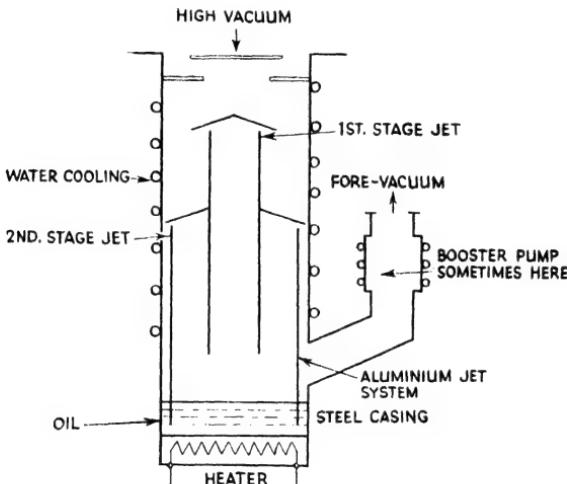


FIG. 31.—Outline of Construction of Large Oil Diffusion Pump.

A steam ejector pump construction is shown in Fig. 32b. A considerable supply of high-pressure steam is necessary. This is often available in large engineering plants. In such circumstances, the steam ejector has many advantages for large-scale vacuum systems where medium low pressures and high pumping speeds are required. This is especially the case in many aspects of chemical engineering practice where water-bearing vapours and corrosive gases are often encountered.

When using oil diffusion pumps care must be taken to ensure that the heating of the oil is correct if a good pumping speed is to

¹ Dushman, S., *Scientific Foundations of Vacuum Technique*, Wiley (Chapman and Hall Ltd.), 1949.

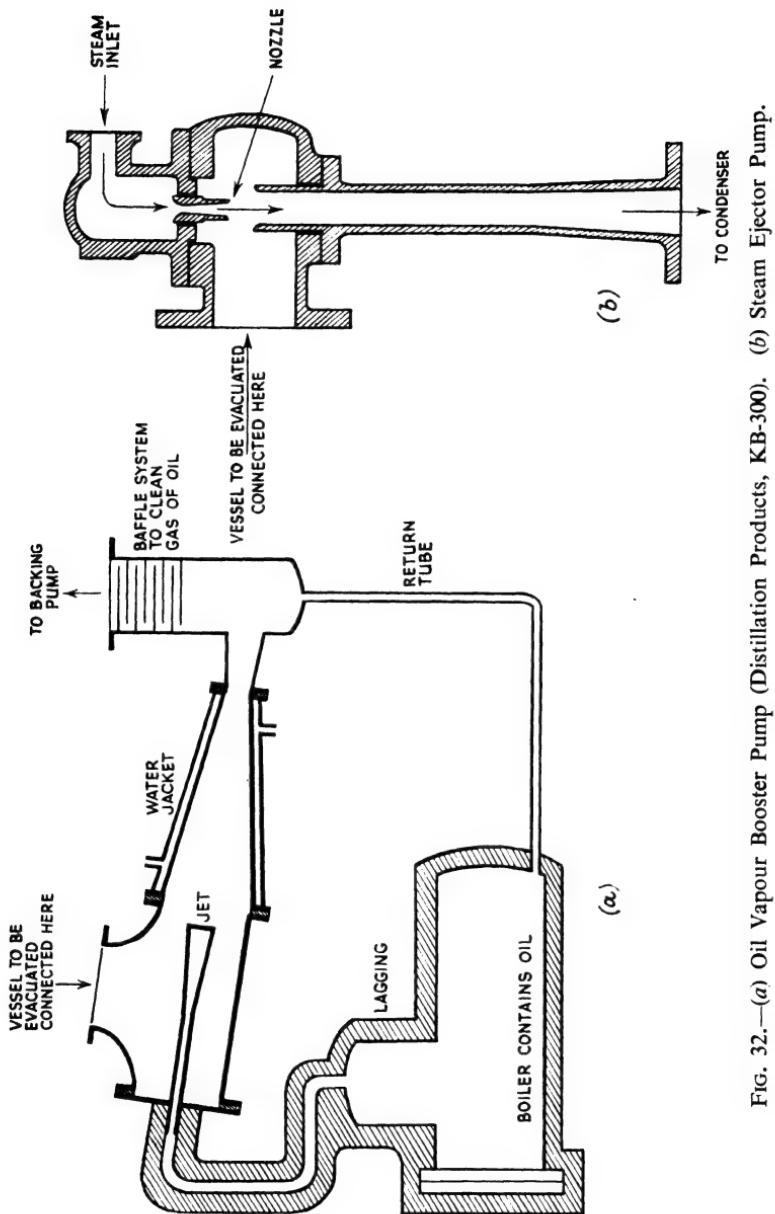


FIG. 32.—(a) Oil Vapour Booster Pump (Distillation Products, KB-300). (b) Steam Ejector Pump.

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be maintained. The temperature is more critical than it is in the case of the mercury diffusion pump. Moreover, the oil must never be heated at a pressure greater than 1 mm. Hg, unless silicone oils are used, otherwise oxidation will occur, and the pump becomes obstructed by an oil of too great a viscosity and vapour pressure.

Pumping Systems. Having described the essential details and working of the various types of pump, it is necessary to discuss their efficient operation.

Fig. 33 is a schematic diagram of a typical pumping system useful for all types of laboratory work. The box pump with air-inlet is at P . T is a pump stand or table erected for convenience of working and to support the diffusion pumps and associated gear.

The backing or box pump should have a pumping speed which is adequate for the diffusion pump used if accumulation of gas in the line between the two pumps is to be avoided. If P_D and P_B are the working pressures of the diffusion and backing pumps respectively, and S_D and S_B are their corresponding pumping speeds, then the simple application of Boyle's law shows that, if the box pump is to extract all the gas supplied to it by the diffusion pump, then S_B must be equal to, or preferably greater than, $S_D \cdot P_D / P_B$. Thus a diffusion pump with a speed of 50 litres/sec. at 10^{-4} mm. Hg requires a backing pump with a speed of $50 \cdot 10^{-4} / 10^{-1}$ or 0.5 litres/sec. at a fore-pressure of 10^{-1} mm. However, because of the restricting effect of the inevitable tubing between the pumps on the flow of gas (see Chapter Three) and particularly using large pumps with demountable systems, or where a leak or evolution of gas is taking place in the vessel, the required backing pump speed, found by this simple calculation, should be multiplied by a factor of five or even ten in practice.

The heating of the diffusion pump Q is performed by an electric heater H . Taps or stop-cocks are arranged at the places indicated so that the vessel may be sealed off from the pump ; the diffusion pump separated from the backing pump ; and the vessel pumped by the backing pump only via tube T_1 , whilst the diffusion pump is still at high vacuum.

A McLeod gauge is attached at M with suitable arrangements for altering its mercury level. A Pirani gauge is joined at R . Chapter Two gives details of operation of these gauges.

X is the tube joining to the vessel undergoing exhaustion. In

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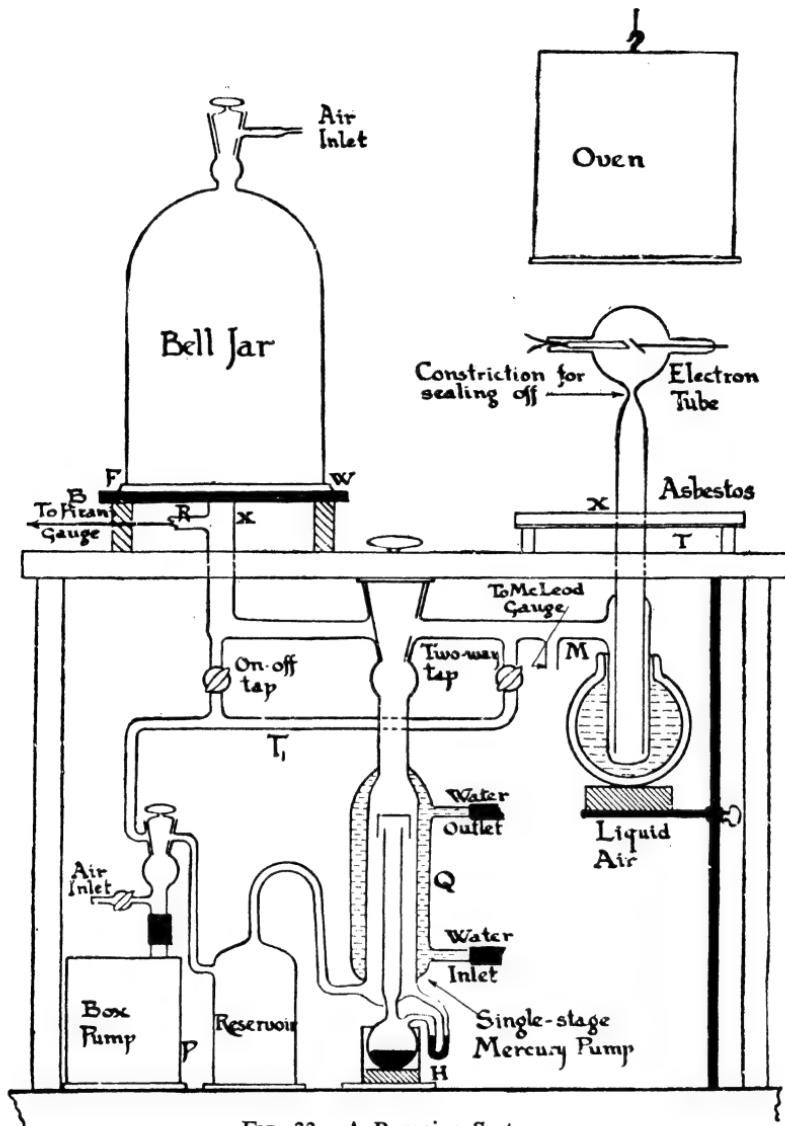


FIG. 33.—A Pumping System.

many types of work (e.g. activation of electron tubes) this will be best directly joined by glassblowing to the vessel. If a demountable system is required, i.e. a vessel which can be readily removed from

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the pump, then a greased or waxed joint¹ is used, as shown at *W*, where a bell-jar is being pumped. *B* is a flat metal or thick glass plate. The bell-jar is seated on the plate by the ground flange *F*. The contact between the flat plate and this flange is either greased with a low vapour-pressure grease, or "Q" compound is spread evenly round the flange periphery at *W*. "Q" compound is a mixture of oils of low vapour pressure and graphite, in appearance much like plasticine. It is marketed by Technical Products Ltd., the makers of the "Apiezon" oils and waxes. The oven shown is lowered over the electron tube being evacuated to give it a "de-gassing" bake before the tube is "activated" (see Chapter Five).

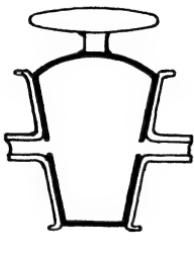


FIG. 34.

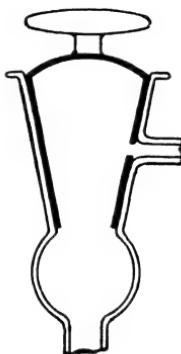


FIG. 35.

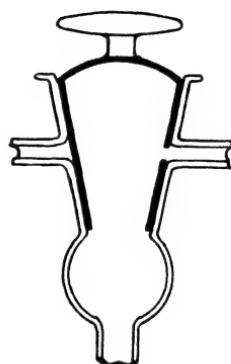


FIG. 36.

Conical joints used in vacuum work have usually either a 1-in-5 or 1-in-10 taper, and are fine-ground by emery, using a steel tool of the correct shape. The female and male joints are separated by a thin smooth layer of grease. A similar procedure is adopted in the case of vacuum taps. Figs. 34, 35 and 36 show various types of taps useful in vacuum work. Wherever possible, it is best to use the types such as those in Figs. 35 and 36 where the stop-cock is under atmospheric pressure, as they are manifestly much less liable to air leaks.

The tap grease used should have as low a vapour pressure as possible. "Apiezon" greases are specially prepared for this purpose (see Chapter Six). Silicone greases are also available and

¹ For most purposes the use of the new "O" ring gaskets is preferable (see page 60).

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manufactured by the Dow Corning Corporation¹ of America. These have remarkably constant viscosity characteristics over the temperature range from -20° C. to 40° C., and very low vapour pressure. At temperatures above 15° C., however, they are more viscous than Apiezon greases so that the stop-cock requires a greater turning torque, but at lower temperatures, between 0° C. and 10° C., they have lower viscosity than the Apiezon greases. They are therefore especially useful in providing readier stop-cock operation after glass vacuum gear has been cold overnight.

If a joint between cones is required which has only to be dismantled at rare intervals, then a hard wax like picein black wax, sealing wax or Khotinsky cement may be used. These are applied



FIG. 37.

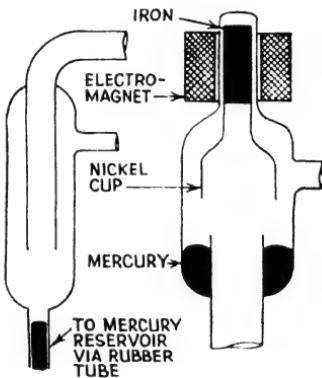


FIG. 38.

FIG. 39.

Cut-offs.

at about 50° C. to the joint, and on cooling a semi-permanent join is effected (see Fig. 37). This procedure is very suitable for a cone joint between glass and metal. The vapour pressures of these various greases and waxes will be found in Chapter Six.

Cut-offs. A mercury "cut-off" is sometimes more convenient than a greased tap. If the mercury is separated from the vessel by the diffusion pump freezing trap, then a "cut-off" is obtained which gives no vapour-pressure troubles.² Two such mercury "cut-offs" are depicted in Figs. 38 and 39.

¹ Available from W. Edwards and Co., Ltd., England.

² G. W. Lobb and J. Bell, *Jour. Sc. Inst.*, **12**, 14, 1935, describe amalgams containing about 0·2% sodium, lithium and bismuth reputed to have vapour pressures as low as 5×10^{-8} mm. Hg at 15° C. for use in a cut-off.

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Fig. 38 shows a type operated by raising a reservoir, whilst Fig. 39 is a magnetically operated type.

Leak Finding. Leak finding in vacuum systems is attempted in a variety of ways :

(1) If possible the parts are immersed in a soap solution and compressed air blown in to indicate the hole.

(2) If the system is a glass one, then the Tesla coil is by far the most useful tool. The system is pumped with the backing pump only, and the Tesla coil probe placed on the glass. A glow discharge is obtained, and if the probe passes over a minute hole in the glass, a spark from the metal probe to the conducting gas will be observed (*see Chapter Two*).

(3) If the leak is probably in a glass-metal joint, or for any other reason cannot be detected by the Tesla coil, then, if a glow discharge is formed in the system, a rag soaked in petrol or methylated spirit passed over the region of the leak will cause a change in the colour of the discharge in that region because some of the organic vapour will pass into the vacuum.

This method only works for leaks of a certain order of size ; if the hole is too small not enough vapour gets in ; if it is too large then the vacuum is undesirably contaminated by too great an inrush of the vapour. It is not greatly recommended.

(4) To find the approximate region of the leak in a complex system involving taps and "cut-offs", the locality of the hole can be found by isolating various parts of the system and giving them "cut-off" tests. The "cut-off" test is the only simple method for a very small leak, i.e. one of the order of a pressure rise from 10^{-6} mm. Hg to 10^{-4} mm. Hg in half an hour.

The vessel is first pumped to the lowest possible pressure and then cut off from the pump for a timed period. The pressure rise during this time is indicated on a gauge. The suspected point of leakage is then covered with hard wax, or thick grease, and another "cut-off" test performed. Repeating the procedure until no rise in pressure is observed after at least a ten-minute "cut-off" finally detects the leak.

A valuable leak-finding method which employs a Pirani gauge with hydrogen as a probe gas is often employed on metal systems. The method is described in the section on Pirani gauges, page 102. The ionization gauge also lends itself to leak detection procedures (*see page 103*).

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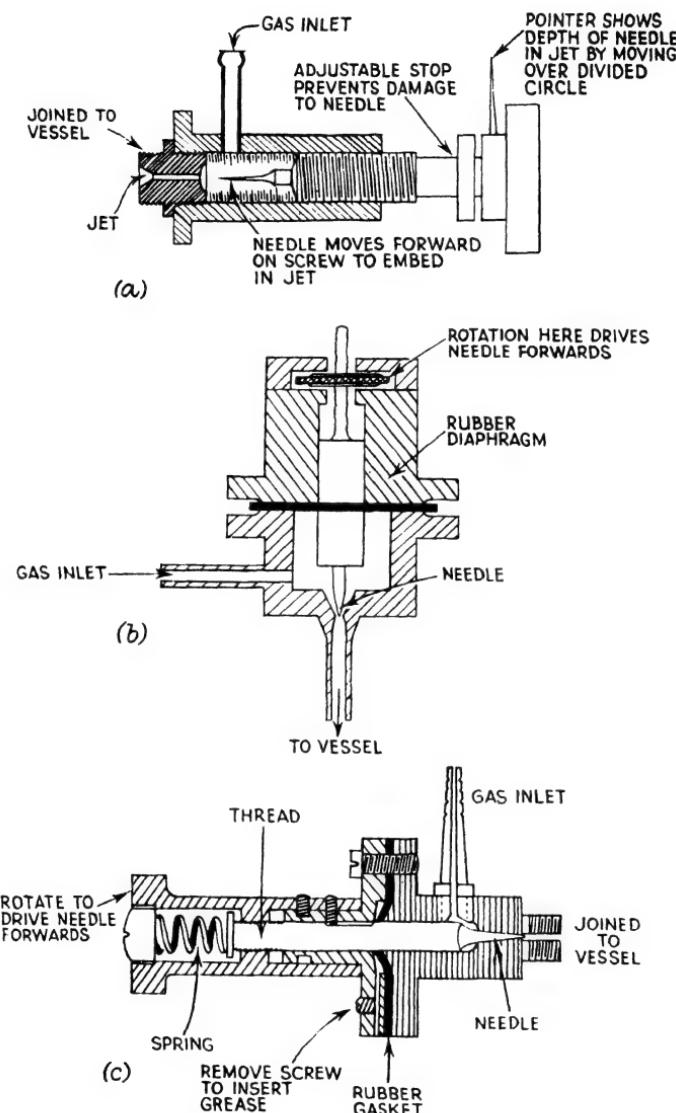


FIG. 40.—Types of Needle Valve.

- (a) Relies on greased fine thread to avoid leak to atmosphere
- (b) Due to P. Alexander, uses rubber
- (c) Manufactured by W. Edwards and Co., Ltd.

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In some classes of work, e.g. evacuating metallic castings, the only means of avoiding leak trouble is to paint heavily the metal so as to fill up any porous regions ; or a thin coating of " Apiezon " W wax can be applied.

Leaks are undoubtedly the vacuum technician's biggest bugbear. An elusive leak may baffle him for some days.

An artificial leak, or regulated leak, can be obtained by means of the needle valves illustrated in Fig. 40.

The Characteristics of Vacuum Pumps.¹ The choice of a backing pump or of a diffusion pump for a particular purpose is best made

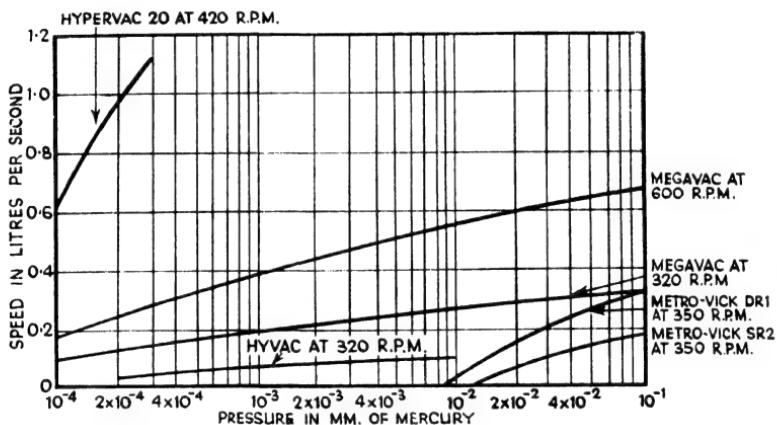


FIG. 41.—Speed vs. Pressure Curves for Some Mechanical Rotary Pumps.²

by considering graphs of the pumping speed plotted against the pressure, or alternatively of the pressure attained against time when a vessel of known size is being evacuated. Such graphs will show both the order of time required for a given exhaust operation, and the lowest possible pressure obtainable.

Fig. 41 gives the performances of the Cenco-Hyvac series of backing pumps. Superimposed on these curves are those of the

¹ This section should be considered in conjunction with the text of Chapters Two and Three on gauges and pumping speed measurements.

² The speeds of the Metro-Vick pumps are recorded using a Pirani gauge (total gas pressure). The other pumps speeds are measured using a McLeod gauge (only air pressure). See Chapter Two, ref. these gauges. (By courtesy of the Metropolitan-Vickers Electrical Co., Ltd.)

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Metro-Vickers DR1 and SR2 pumps. Apparently these latter pumps have the inferior performance; in practice there is little to choose between the two manufacturers' types. The discrepancy shown by the curves is due to the use of a McLeod gauge in obtaining the figures for the Cenco-Hyvac pumps, whereas a Pirani gauge was used for recording the performances of the Metro-Vickers types.

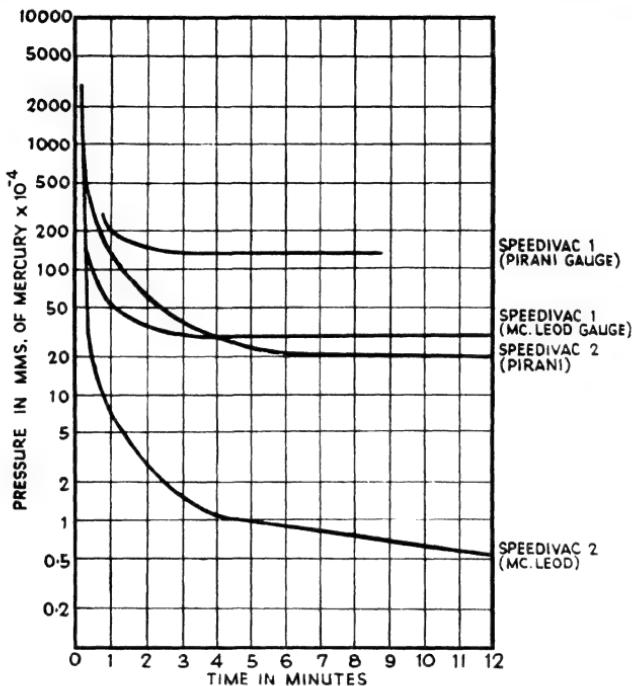


FIG. 42.—Pressure-time Characteristics of Speedivac Pumps.

As is explained in Chapter Two, the McLeod records only the pressures due to the so-called perfect gases, whereas the Pirani gauge records the total gas pressure, including that due to any condensable vapour such as water vapour. Bearing in mind that the mechanical pumps cannot cope with condensable vapours, it will be appreciated why the performances seem inferior when a Pirani is used. Often the total gas pressure reading is the more important record of the two, since water vapour, at least, is commonly present in systems.

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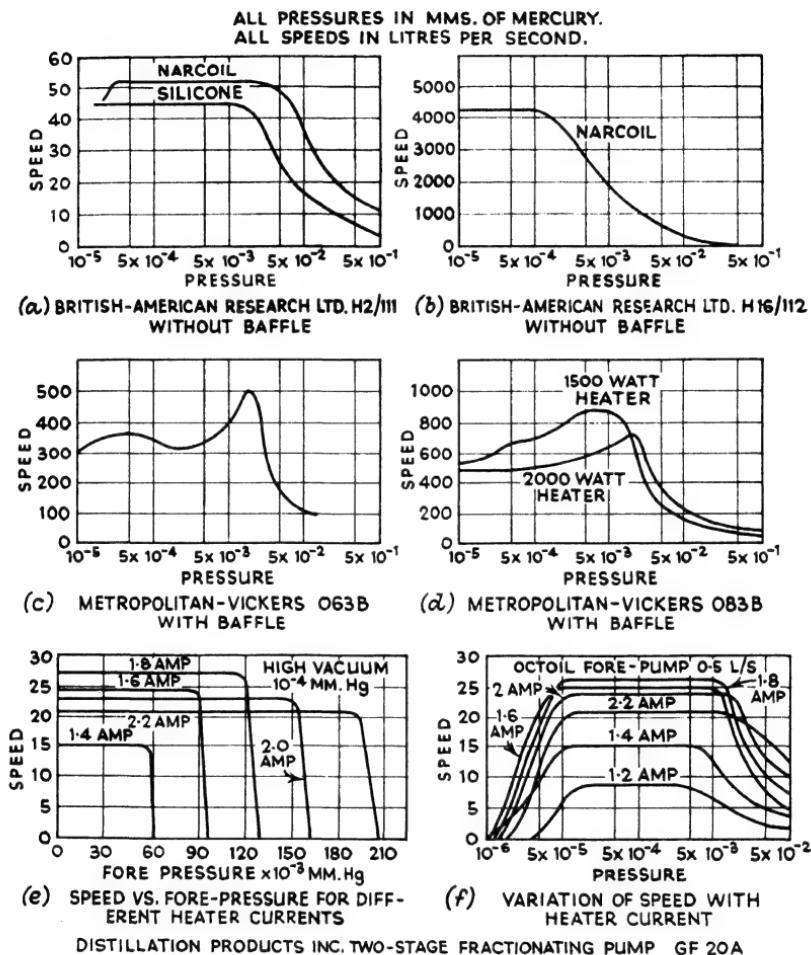


FIG. 43.—Characteristics of Oil-diffusion Pumps.

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A further indication of the disparities in readings obtained with these two gauges is shown by Fig. 42. This shows the pressure versus time curves for the Speedivac pumps.¹ A direct connexion to a gauge of 100 cc. capacity was made, and in the case of both types 1 and 2, two curves were plotted, one using the McLeod, the other using a Pirani gauge. Thus using the Speedivac 2 pump, a two-stage model, the pressure recorded after five minutes was 8×10^{-5} mm. Hg according to the McLeod, but was as high as 2.8×10^{-3} mm. Hg if a Pirani gauge was used. Which graph is considered depends on the circumstances prevailing, e.g. is a diffusion pump used as well as a mechanical pump, is there a drying agent, or does the system incorporate a freezing trap?

Fig. 43² shows various characteristics of some oil diffusion pumps. (a) compares the speeds of the same 2" diameter inlet pump using, firstly, Narcoil 10 and secondly, D.C. silicone oil with the same heater watts. The speed using silicone is some 16% lower because of its higher boiling-point; (b) gives the characteristics of a 16" diameter inlet pump without baffle; (c) shows the characteristic for a 6" diameter pump with baffle which has a peak speed at 2×10^{-3} mm.; (d) indicates the effect of increased heater wattage on a baffled 8" diameter inlet pump, the increase causing the speed to fall off at the lower pressures but rise at the higher pressures; (e) and (f) show how the fore-pressure and the heater current affect the speed of a two-stage glass fractionating pump.

DATA ON MANUFACTURERS' PUMPS

An outline of the characteristics of the various pumps available from British and American manufacturers is given in the following tables.² The displacement quoted for mechanical rotary pumps is the volume of air passed by the pump per unit time at atmospheric pressure. The displacement is considerably greater than the pump speed at a low pressure; though a useful indication of a pump's performance yet, as a figure of merit, it should be accepted with some reserve. In all cases, a McLeod gauge is used for pressure measurements (see Chapter Two). Speeds are given in litres per minute. One cubic foot = 28.3 litres.

¹ By courtesy of W. Edwards & Co., Ltd. The two model numbers quoted are now obsolete.

² The data given is by courtesy of the various manufacturers concerned.

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MECHANICAL ROTARY PUMPS WITH GAS BALLAST

Pump	No. of stages	Free air displace- ment in litres/min.	Limiting pressure in mm. Hg		H.P. of motor required
			Full gas ballast	Ballast off	
<i>W. Edwards & Co., Ltd. (British)</i>					
2SC20	2	22	10^{-3}	10^{-3}	$\frac{1}{2}$
1SC30	1	32	1	2×10^{-2}	$\frac{1}{2}$
1SC50	1	48	1	10^{-2}	$\frac{1}{2}$
2SC50	2	48	5×10^{-3}	10^{-4}	$\frac{1}{2}$
1SC450	1	450	1	10^{-2}	1
1SC1300	1	1325	2	10^{-2}	2
1SC3000	2*	3120	2	10^{-2}	5
<i>E. Leybold's Nachfolger (German)</i>					
VP2	1	34	5	5×10^{-2}	$\frac{1}{2}$
S2	1	34	1	2×10^{-3}	$\frac{1}{2}$
D2	2	34	5×10^{-2}	10^{-5}	$\frac{1}{2}$
VP5	1	85	5	5×10^{-2}	$\frac{1}{2}$
S5	1	85	1	2×10^{-3}	$\frac{1}{2}$
D5	2	85	5×10^{-2}	10^{-5}	$\frac{1}{2}$
VP10	1	170	5	5×10^{-2}	$\frac{1}{2}$
S10	1	170	1	2×10^{-3}	$\frac{1}{2}$
D10	2	170	5×10^{-2}	10^{-5}	1
D25	2	425	5×10^{-2}	10^{-5}	$2\frac{1}{2}$
S50	1	820	1	2×10^{-3}	3
S150	1	2490	1	2×10^{-3}	4
S300	1	5000†	1	2×10^{-3}	8
<i>National Research Corp. (American)</i>					
NRC2S	1	35	8×10^{-1}	2×10^{-3}	$\frac{1}{2}$
NRC4S	1	108	2×10^{-1}	2×10^{-3}	$\frac{1}{2}$
NRC6S	1	190	4×10^{-1}	2×10^{-3}	$\frac{1}{2}$
NRC15S	1	417	4×10^{-1}	2×10^{-3}	1
NRC30S	1	835	4×10^{-1}	10^{-2}	$1\frac{1}{2}$
NRC100S	1	2830	10^{-1}	10^{-2}	5
NRC200S	1	5700	4×10^{-1}	10^{-2}	10
NRC400S	1	11,400	1	10^{-2}	20
NRC2D	2	35	5×10^{-2}	10^{-5}	$\frac{1}{2}$
NRC4D	2	94	10^{-2}	10^{-5}	$\frac{1}{2}$
NRC6D	2	212	2×10^{-3}	10^{-5}	$\frac{1}{2}$
NRC15D	2	417	8×10^{-3}	5×10^{-5}	1

* 2 stages in parallel.

† S600 = two S300's in parallel.

MECHANICAL ROTARY PUMPS

Pump	No. of stages	R.P.M.	Free air displacement litres/min.	10^{-1} mm. 10^{-2} mm.	Pumping speed in litres/min. at	Limiting pressure \dagger in mm. Hg	H.P. of motor required
<i>Central Scientific Co. (American)</i>							
Hyvac.	2	345	10	6	5.4	3×10^{-4}	$\frac{1}{4}$
Megavac	2	325	31	18	16.2	10^{-4}	$\frac{1}{3}$
Megavac	2	600	57	39.6	32.4	10^{-4}	$\frac{1}{4}$
Hypervac 23	2	510	240	—	—	5×10^{-3}	$\frac{1}{4}$
Hypervac 25	2	570	264	175	150	10^{-4}	$\frac{1}{4}$
Hypervac 100	2	450	960	720	600	10^{-4}	$\frac{2}{3}$
Pressovac 4 *	1	600	35	30	—	5×10^{-3}	$\frac{1}{4}$
<i>W. Edwards & Co., Ltd. (British)</i>							
1 SP 30 *	1	700	32	20	—	2×10^{-2}	$\frac{1}{4}$
1 S 50	1	450	48	33	9	5×10^{-3}	$\frac{1}{4}$
1 S 150	1	450	144	78	18	5×10^{-3}	$\frac{1}{3}$
1 S 450A	1	350	450	240	60	5×10^{-3}	$\frac{1}{1}$
2 S 20	2	450	22	18	13.8	10^{-4}	$\frac{1}{4}$
2 S 50	2	450	48	40	25.8	5×10^{-3}	$\frac{1}{3}$
2 S 150	2	450	144	103	75.0	10^{-4} to 5×10^{-3}	$\frac{1}{4}$
<i>Kinney Mfg. Co.† (American)</i>							
CVD556	2	525	420	330	270	10^{-4}	1
CVD8610	2	500	1,270	935	800	10^{-4}	3
VSD556	1	450	360	220	82.5	5×10^{-3}	$\frac{1}{4}$
VSD778	1	360	745	440	193	5×10^{-3}	$\frac{1}{2}$
VSD8811	1	360	1,290	800	330	5×10^{-3}	2
DVD8810	1	450	3,040	1,920	740	5×10^{-3}	5

50

DVD12814	.	.	1	415	6,000	3,600	1,500	5×10^{-3}	10
DVD14918	.	.	1	360	8,500	5,250	2,060	5×10^{-3}	15
DVD141418	.	.	1	360	13,350	8,250	3,440	5×10^{-3}	25
DVD181420	.	.	1	360	19,300	11,800	4,950	5×10^{-3}	40
<hr/>									
<i>Metropolitan-Vickers Ltd.</i>									
(British)									
SR2 or S4	.	.	1	475	24	15	5.2	5×10^{-3}	$\frac{1}{2}$
DR1	.	.	2	360	57.6	40	20	10^{-4}	$\frac{1}{2}$
SR3 or S59	.	.	1	600	390	330	—	10^{-3}	1
SR5 or S330	.	.	1	455	1,788	780	—	10^{-2}	3
<hr/>									
<i>Stokes</i> § (American)									
Microvac146E	.	.	1	350	282	192	126	2×10^{-3}	1
146E	.	.	1	500	426	288	192	2×10^{-3}	1
148E	.	.	1	370	900	618	408	2×10^{-3}	$\frac{1}{2}$
149E	.	.	1	385	1,698	1,152	762	2×10^{-3}	3
212E	.	.	1	385	3,240	2,208	1,458	2×10^{-3}	5
412E	.	.	1	400	6,660	4,530	3,000	2×10^{-3}	$\frac{1}{2}$
612E	.	.	1	425	14,280	9,720	6,420	2×10^{-3}	25
<hr/>									
<i>Welch Sc. Co. (American)</i>									
Duo-seal 1400	.	.	2	450	21	12	10	10^{-4}	$\frac{1}{2}$
1405	.	.	2	300	33.4	23	20	5×10^{-5}	$\frac{1}{2}$
1405	.	.	2	525	58.2	39.6	36	5×10^{-5}	$\frac{1}{2}$
1406	.	.	1	300	33.4	22.8	12	5×10^{-5}	$\frac{1}{2}$
1403	.	.	1	375	100	78	34.8	5×10^{-5}	$\frac{1}{2}$
1404	.	.	1	300	33.4	—	—	2×10^{-2}	$\frac{1}{2}$
1410	.	.	1	450	21	—	—	2×10^{-2}	$\frac{1}{2}$
1395	.	.	1	1725	12	—	—	2×10^{-2}	$\frac{1}{2}$
1397	.	.	2	300	240	200	—	10^{-4}	$\frac{1}{2}$

* Also usable as a compressor.

† The limiting pressure, or ultimate vacuum, is the manufacturers' guaranteed figure; the true limit can be lower than this value.

‡ Also available from General Engineering Co., Ltd., Radcliffe, Lancs., England.

§ From H. M. Sullivan, *Rev. Sc. Inst.*, **19**, 1, 1948.

DIFFUSION AND EJECTOR PUMPS OF DISTILLATION PRODUCTS, INC. (AMERICA).

Type	No. of stages	Fluid charge	Speed in litres/sec.	Ultimate vacuum in mm. Hg	Heater, watts	Inlet port, dia. in.	Max. backing pressure, mm. Hg
<i>Boosters</i>							
MB100	.	1	225 gm. Butyl * phthalate	100 at 10^{-3} mm. Hg	5×10^{-5}	250-500	3 $\frac{1}{8}$ 0.3
MB200	.	1	600 gm. phthalate	200 , , , , , ,	5×10^{-5} 10^{-4} 10^{-4}	200-1080 275 300	6 0.4
VMB1	.	1	55 c.c. phthalate	1			— 0.5
VMB7	.	1	100 c.c.	"			— 0.5
VKB8	.	1	700 gm. Myvane	7 at 7×10^{-2} mm. Hg	1.5×10^{-3}	815	14 0.5
VKB150	.	1	2 gal.	150 at 10^{-1} mm. Hg	10^{-3}	2400-3000	6 0.4
KB300	.	1	5 $\frac{1}{2}$ gal. K.B. fluid	350 at 2×10^{-1} mm. Hg	10^{-2}	5000-10000	6 3.5
GB3 (Pyrex-glass)	.	2	150 c.c. Butyl sebacate	3 at 10^{-1} mm. Hg	5×10^{-5}	35-160	— 1.0
<i>Metal Diffusion</i>							
MC275	.	1	200 gm. Octoil †	275 at 10^{-4} mm. Hg	5×10^{-6}	250-450	3 $\frac{1}{8}$ 0.1
MC500	.	1	600 , , , , , ,	500 , , , , , ,	"	330-1080	6 0.1
MC3000	.	1	2500 , , , , , ,	3000 at 7×10^{-4} mm. Hg	"	1850	14 0.1
<i>Metal Fractionating</i>							
VMF2	2	35 gm. Octoil ‡		2 at 10^{-4} mm. Hg	10^{-6}	85	$\frac{15}{16}$ 0.1
VMF5 or VMF10 water-cooled	2	35 , , , , , ,		5 , , , , , ,	"	85	$\frac{15}{16}$ 0.1
	2	55 , , , , , ,		10 , , , , , ,	"	135	$\frac{15}{16}$ 0.1
VMF20	2	100 , , , , , ,		20 , , , , , ,	"	200	$\frac{15}{16}$ 0.1

VMF50	2	150	"	"	"	"	"	325	0·5
VMF100	2	300	"	"	"	"	"	375	0·5
VMF200	3	300	"	"	250 at 3 $\times 10^{-5}$ mm. Hg	4×10^{-7}	310-375	0·1	
MCF700	2	500	"	"	700 " 3 $\times 10^{-4}$ "	5×10^{-7}	800	0·1	
MCF1400	2	1000	"	"	1600 at 10 $^{-3}$ mm. Hg	10^{-6}	2100-2700	0·25	
MCF5000	3	1½ gal. Myvane		5000	8×10^{-7}		3000	0·1	
<i>Mercury Diffusion</i>									
GHG10	2	80 c.c. Mercury	6				250	2	
Pyrex	2	110	"	6			350-500	5	
GHG105	3	160	"	20			400-500	10	
Glass	2	190	"	50			500	0·4	
MHG50 (steel)									
<i>Glass-metal Diffusion</i>									
GM220AB	2	200 gm. Octoil †	220 at 10 $^{-4}$ mm. Hg	10^{-6}	130-325	4	0·15		
glass (air)									
GM220WB	2	200	"	"	"		130-325	4	0·15
glass (water)									
GM220WB	2	200	"	"	"				
min- ium (water)									
alu- mum jets									
<i>Glass Fractionating</i>									
G4 (air)	1	65 g. Octoil †	4 at 10 $^{-4}$ mm. Hg	10^{-5}	25-50	1½	0·08		
GF5A (air)	2	50	" ‡	$3-5 \times 10^{-7}$	40-80	½	0·1		
GF20A or W (air or water)	2	130	"	"	7×10^{-7}	90-260	0·1		
GF25A (air)	3	200 gm. Octoil S	25	"	5×10^{-8}	90-250	0·1		
GF25W (water)	3	200	"	"	"	90-250	0·1		

* Or butyl sebacate.

† Or Arnol S.

‡ Or preferably Octoil S.

HIGH VACUUM TECHNIQUE

Pumps made by Pulsometer Engineering Co., Ltd. For systems where only a moderate vacuum is required, the fast-running (FR) pumps are useful and economical. Directly coupled to an electric motor, they are available in three sizes, as follows :

Pump size	R.P.M.	Displacement in litres/min.	Limiting pressure	H.P. of motor
1"	1425	27.5	0.1 mm. Hg	$\frac{1}{6}$
3"	1425	385	25" *	1
5"	1250	688	25"	2

* A rough vacuum is often specified by engineers in terms of the mercury rise it will produce in a manometer. Thus 25" is 5" off a perfect vacuum—which would produce a manometer rise of 30". Hence, $25'' \equiv 30'' - 25'' \equiv 12.7$ cm. Hg.

Note. The 3" pump will produce a vacuum of 16" ($\frac{1}{2}$ atmosphere approx.) against a $\frac{3}{16}$ " diameter leak. The 5" pump will do likewise against a $\frac{5}{16}$ " leak.

Pulsometer Geryk Pumps

Type No.	No. of stages	Displacement in litres/min.	Limiting pressure, mm. Hg	H.P. of motor
4ST1	1	110	10^{-2}	$\frac{1}{2}$
3"	1	68	10^{-2}	$\frac{1}{2}$
4"	1	170	10^{-2}	$\frac{3}{4}$
6"	1	550	10^{-2}	$1\frac{1}{2}$
6/20	1	566	5×10^{-3}	2
8"	1	1090	10^{-2}	2
8/50	1	1700	5×10^{-3}	3
10	1	2125	5×10^{-3}	$5\frac{1}{2}$
12	1	2480	5×10^{-3}	6
2S1 *	1	68	5×10^{-3}	$\frac{1}{2}$
4S1	1	136	5×10^{-3}	$\frac{1}{2}$
2D1 †	2	68	10^{-5}	$\frac{1}{2}$
4D1 ‡	2	136	10^{-5}	$\frac{1}{2}$

* A 2S3 and 2S6 are also available ; these are respectively three and six 2S units in parallel.

† A 2D3 is three 2D1 units in parallel, giving three times the speed.

‡ A 4D3 and a 4D5 are with three and five 4D1 units in parallel respectively.

Diffusion-Condensation and Ejector Pumps. Pumping speeds are here quoted in litres per second.

THE PRODUCTION OF HIGH VACUA

*British American Research Ltd.*¹ (British). The oil diffusion pumps detailed all give an ultimate vacuum of 10^{-6} mm. Hg or better, using Narcoil 10, type 4101 pump fluid, D.C. silicone oil 702 or 703, or Apiezon B oil, provided the recommended backing conditions are observed. The speed of the pump will depend on the oil used and the heater wattage; thus using Narcoil or Apiezon B, the pump will be some 20% faster than if D.C. silicone is used with the same heat input, because the silicone oils have higher boiling points. The speed figures given are for Narcoil, and without a baffle present.

Type	Fluid charge, c.c.	Speed in litres/sec.	Heater, watts	Inlet port, dia. in.	Max. backing pressure, mm. Hg	Backing pump speed litres/min.
H2/111	80	50 at 10^{-3} mm. Hg	220	2	0.2	96
H6/104	400	570 " " "	750	6	0.2	700
H10/108	1000	1240 " " "	2250	10	0.45	1400
H16/112	2500	1830 " " "	2250	16	0.25	1400
B6/107	3 gal.	4100 at 10^{-4} mm. 1050 at 10^{-2} mm.	6000	6	0.9	2750

Note. The B6/107 is a booster pump designed to have a high speed at a pressure of 10^{-2} mm. Hg and operate with a high backing pressure. It is valuable in having a large pumping speed in the range where ordinary oil-diffusion pumps and mechanical rotary pumps have low speeds.

Distillation Products, Inc. (American). The pumps listed on page 52 are water-cooled, and made with steel jackets and aluminium jets, unless otherwise specified.

W. Edwards & Co. Ltd. (British). Oil diffusion pumps listed use D.C. silicone or Apiezon oils. The ultimate vacuum is $< 5 \times 10^{-6}$ mm. Hg. They are all fabricated using steel casings and aluminium jet systems.

Mercury diffusion pumps are also supplied. Using a liquid air trap, they give an ultimate vacuum better than 10^{-6} mm. Hg. These pumps are all steel except the GM2, which is made of borosilicate glass.

¹ Now known as *Vacuum Industrial Applications Ltd.*

HIGH VACUUM TECHNIQUE

Type	No. of stages	Oil charge, c.c.	Speed at 10^{-4} mm. Hg in litres/sec.	Heater, watts	Inlet port, dia. in.	Max. backing pressure, mm. Hg	Backing pump speed, litres/min.
102	2	10	11	175	1	0.5	20
203	3	50	70	250	2	0.5	50
403	3	100	325	500	4	0.5	150
603	3	200	625	1350	6	0.5	250
903B	3	300	1500	2250	9	0.5	450
1605	4	2000	5500	5200	16	0.5	

Note. The types 403, 603 and 903B incorporate an integral booster third stage. The 1605 has a booster fourth stage. Baffles which prevent oil vapour getting into the system are available for each type of pump. Such baffles reduce the speed. The F203 pump is a development of the type 203; it has a self-purifying action and gives an ultimate vacuum of 5×10^{-7} mm. Hg.

Type	No. of stages	Mercury charge, c.c.	Speed at 10^{-4} mm. Hg in litres/sec.	Heater, watts	Inlet port, dia. in.	Max. backing pressure, mm. Hg	Backing pump speed, litres/min.
1M2	2	15	10	175	1	1.0	20
1M2	2	30	10	350	1	4.0	30
GM2	2	50	13	150	1½	1.0	20
2M2	2	50	75	350	2	0.35	50
2M4	4	200	75	850	2	30	—
6M3	3	200	625	1300	6	0.5	250

Oil ejector pumps of the booster type are also available from this firm. These have peak speeds in the region of 10^{-1} to 10^{-2} mm. Hg and work against backing pressures of the order of 2 to 3 mm. Hg. They are intended as intermediaries between large oil diffusion pumps and mechanical rotary pumps, or to provide high pumping speeds at intermediate vacua where the speed of a mechanical rotary pump is low.

Type	Ultimate vac., mm. Hg	Peak speed, litres/sec.	Max. backing pressure, mm. Hg	Backing pump speed, litres/min.	Oil charge, c.c.	Heater, max. watts
9B1	$< 10^{-4}$	40 at 1.85 kW	2-5	450	1500	2.25 kW
9B3	$< 10^{-4}$	850 at 2.25 kW	2-3	450	1500	2.25 kW
18B3	$< 10^{-4}$	1300 at 6.4 kW	2-3	2800	10,000	6.4 kW

Types 9B1 and 9B3 require a warming-up time of 25 mins. This time, for type 18B3, is 60 min.

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National Research Corporation (American)

Type	Fluid charge	Max. speed, litres/sec.	Heater, watts	Max. backing pressure, mm. Hg	Backing pump capacity, litres/min.	Ultimate vac., mm. Hg
H-2-P	85	70	200	0·18	85	$1\cdot5 \times 10^{-6}$
H-4-P	250	300	500	0·25	200	1×10^{-6}
H-4-SP	250	300	500	0·25	200	$1\cdot5 \times 10^{-6}$
H-6-P	400	730	950	0·20	850	$1\cdot5 \times 10^{-6}$
H-10	1000	1400	2250	0·45	1400	5×10^{-5}
H-16-P	800	4000	4050	0·23	2800	4×10^{-6}
B-1	25	8·5	85	0·20	28	4×10^{-4}
B-2	100	72	450	1·0	230	$1\cdot5 \times 10^{-4}$
B-4	375	200	1380	0·9	340	2×10^{-4}
B-6	3 gal.	520	6000	0·9	2800	$2\cdot5 \times 10^{-4}$

These oil-diffusion pumps employ Narcoil. Type numbers preceded by the letter H are for diffusion pumps, whereas the letter B denotes a booster pump.

Metropolitan-Vickers Electrical Co., Ltd. (British). The oil diffusion pumps listed are operated with Apiezon oils.

Type	Fluid charge	Speed at 10^{-3} mm. in litres/sec.	Heater, watts	Inlet port, dia. ins.	Max. backing pressure, mm. Hg	Backing pump capacity, litres/min.	Ultimate vac., mm. Hg
02	50 c.c. Apiezon A	7	450	1 $\frac{1}{4}$	0·35	20	10^{-5}
03B	75 c.c. Apiezon B	30	600	2 $\frac{1}{8}$	0·35	30	10^{-6}
043B		250 at 10^{-4} mm. Hg		4	0·2		
063B	725 c.c. Apiezon G	400	1150	6	0·1	200	10^{-6}
083B	1350 c.c. Apiezon G	500	1900	8	0·12	250	10^{-6}
0203B	5 gal. Apiezon G	3100	3400	12	0·03	Use 083B	10^{-6}

Note. The speeds quoted in these cases are with baffles. A baffle will reduce the speed by about 40%.

The Construction of Vacuum Systems.¹ Glass is the most useful material for the construction of small vacuum systems. If, however, a vessel to be used has a maximum surface dimension of

¹ See Chapter Six for the properties of the materials discussed in this section.

HIGH VACUUM TECHNIQUE

greater than twelve inches then, in most cases, it is best constructed of metal since the atmospheric pressure of fifteen pounds per square inch renders flat glass surfaces of any extent liable to implosion. Such a danger can be partially avoided by increasing the thickness of the glass used, by choosing a glass of great tensile strength and with heat-resisting properties like Pyrex, and by so rounding the glass surface that the forces acting on it due to external air pressure are not directly additive. When a flat surface in glass of a square foot separates a vacuum from the atmosphere, then the resultant force on it is $144 \times 15 = 2160$ pounds weight, almost a ton ! Obviously much care is needed in designing a system which has to withstand such a stress.

The most commonly used metals for constructional purposes are copper, brass, gun-metal, drawn and cast steel, and aluminium. Which metal is chosen depends primarily on its likelihood to be attacked chemically by active materials in the vacuum, thus copper cannot be used if mercury is present. Otherwise the choice depends on the readiness of working, and the porosity of the metal. In this latter connexion tin-plating, painting with shellac, or even coating with "W" wax are useful techniques.

The advantages of glass over metal in vacuum systems are :

- (a) Glass can be heated to just below its melting point to rid it of occluded gases and vapours.
- (b) Electrode assemblies inside glass envelopes can be readily heated by eddy currents at high frequency (*see page 128*).
- (c) Glass is non-porous.
- (d) A leak can be very readily found using a Tesla coil.
- (e) Intricate shapes in glass can be fashioned by a skilled glass-blower. Also selected glasses can be joined together directly, or if of different coefficients of expansion, by using intermediary glasses in a graded seal. Wires of tungsten, copper, platinum and also of nickel alloys can be sealed into glass (*see page 182*).
- (f) The transparency of glass, enabling structures in vacuum to be observed, is a valuable feature in many circumstances.

In general it is much better to use a heat-resisting borosilicate glass like Pyrex or Hysil rather than a soda glass in vacuum work.

Often in vacuum work a joint is needed between two materials

THE PRODUCTION OF HIGH VACUA

which cannot be directly fused together. Indeed, the selection of materials to be used, and the design of the apparatus, are often decided by this factor. The discovery of some new technique for sealing two different materials together has, on many occasions, led to the design of new apparatus, for example, in the case of the all-metal radio valves. The nature of the join to be effected is influenced by the degree of permanency required, and whether the join will have to withstand abnormal temperatures. Demountable joins are best made by greased conical or flat unions between glasses and metals, or a semi-permanent junction can be made using a hard wax like picein, or W-wax, applied at about 50° C.

Two metal or glass tubes of the same diameter can be satisfactorily joined by enclosing in a thin rubber hose tubing, coated over with shellac. Plastic varnishes, employing some solvent carrier material, are useful in this case. A temporary, but not greatly recommended, join between two tubes can be made if one slides closely inside the other, and the join is covered with "Q" compound.

A union between two tubes of greater diameter than 2", or between a vessel and a flat base plate can be made using a rubber gasket (*see Fig. 44*). A system which is being continually opened for the insertion of new specimens or apparatus is most conveniently made in this manner. The alternatives are a greased joint, which is messy for repetition work where the vessel is being frequently re-evacuated, or to smear "Q" compound round the outside of a flange, as in the bell-jar seated on a flat plate as shown in Fig. 33.

A rubber gasket needs to undergo uniform deformation under the pressure exerted on it by the components of the joint (e.g. bell-jar and base-plate) if it is to give a satisfactory seal free from air-leaks. Thus the gasket shown in Fig. 44a must needs be flattened and squeezed out laterally to some extent by the pressure on it. In this connexion the selection of the width of the annular gasket is important. If this width is decreased, then the air-path from the atmosphere to the vacuum in the vessel is reduced, so that leakage is more likely. On the other hand, the gasket will have the same total pressure on it due to the atmosphere acting via the cover lid irrespective of the gasket width; hence, if this width is reduced, the compression force on the rubber is increased, since the pressure exerted is constant, the gasket area is less, and pressure is force per unit area. This greater compression of the rubber will give a more

HIGH VACUUM TECHNIQUE

satisfactory seal. A narrow gasket is therefore preferable, provided it is not so narrow ($<\frac{1}{4}$ ") as to give a risk due to reduce air path. In many ways, the best solution is the "O" ring gasket made of rubber or, preferably, oil-resisting neoprene cord, of circular cross-section, since the compressive force is then initially

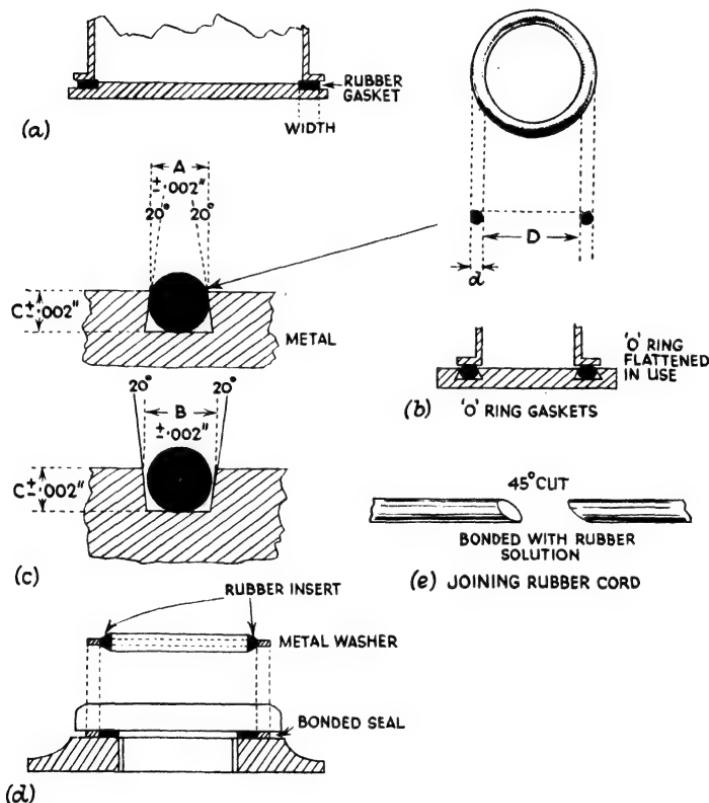


FIG. 44.—Use of Rubber Gaskets,

over a very small area before the circular section is deformed to an "elliptical" shape (Fig. 44b). Moreover, mounted in slots set in a metal plate, such "O" gaskets expose a minimum of rubber to the vacuum. The use of these rubber rings is best arranged in accordance with the machining dimensions indicated in Fig. 44c, in conjunction with the following table :

Dia- meter <i>d</i>	Dimensions of "O" rings available ¹					Groove dimensions		
	Internal diameter <i>D</i>			"O" ring section diameter <i>d</i>		A	B	C
0.070	0.125		0.375			0.07	0.059	0.095
0.103	0.187	0.312	0.563	0.625	0.875	0.937	0.103	0.088
0.139	0.062	1.250	1.375	1.500	1.625	1.750	2.000	2.187
0.210	3.000	3.250		4.375		5.125		5.875
0.275	6.375			6.875				0.275

All dimensions in inches.

¹ Courtesy of W. Edwards and Co., Ltd. "O" rings also available from Linear Inc., Philadelphia, who supply silicone rubber-like material capable of withstanding higher temperatures.

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The use of a lead wire gasket is illustrated by Fig. 45. More care is needed in this case to ensure a correct seating of the gasket in the type of groove illustrated ; such a lead gasket will not effect a vacuum-tight join of the type shown in Fig. 44a, whereas rubber serves satisfactorily. Fig. 46 indicates a method of inserting a slideable metal rod into a vacuum chamber, the leak-free join depending on a rubber gasket in which the hole is smaller than the rod diameter so that the rubber is displaced, as shown, to form a cone-shaped union.¹

More specialized types of junctions between materials which are useful in many circumstances are :

(a) Metal can be soldered to glass or porcelain by metallizing the glass with platinum and soldering to this. A more satisfactory

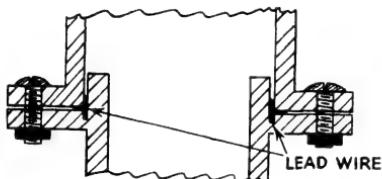


FIG. 45.—Use of a Lead Wire Gasket.

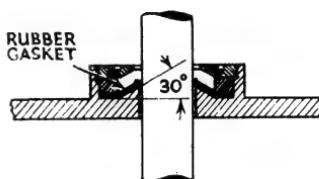


FIG. 46.—The Wilson Seal.

procedure is to use silver to coat the glass, in which case thicker metal films of greater adhesion can be obtained (S. T. Martin²). The silver coating is produced by firing a coating of silver oxide in a carrier at 600° to 700° C. on the end of a piece of Pyrex tube, suitably fire-polished. The silver deposit is carefully dried when firing, and afterwards burnished. The soldering flux consists of a moderately concentrated solution of tartaric acid in water to which has been added a third of its volume of glycerine. This is spread on the work, and the soldering iron, using 60 tin-40 lead solder, is directly applied. The metal tubing used must have a thin wall (10^{-2} cm.) at the join to allow it to stretch under the expansion and contraction so that the glass tube is not fractured.

(b) Housekeeper joints between copper and glass tubing can be made in sizes up to 10 cm. in diameter. The copper has a much larger coefficient of heat expansion than the glass, which is com-

¹ See R. R. Wilson, *Rev. Sc. Inst.*, **12**, 91, 1941.

² S. T. Martin, *Rev. Sc. Inst.*, **11**, 205, 1940.

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monly Pyrex or soda-glass, but the high ductility of the copper enables the differential expansions and contractions which occur to be taken up by the deformation of the copper. Seals between special glasses and iron-nickel-cobalt alloys (Fernico) have been similarly effected.

(c) Porcelain can be directly sealed to Pyrex tubing if the diameters employed are not greater than half an inch. Quartz may be sealed to Pyrex using silver chloride as a cementing material, necessitating fusion at 450° C.

(d) Mica can be sealed to glass or metal, making small flat mica windows on a vessel possible. A low-melting-point lead borosilicate glass is used as a cement (J. S. Donal¹). The mica window cannot comfortably be more than 3 cm. in diameter. It is made slightly larger than the glass tube end over which it is placed. The low melting-point glass is ground fine, mixed into a paste with water, and painted on to the tube end, which must be ground. The mica is pressed on to this preparation, and the paste spread also round the edges of the mica to prevent them splitting. Heating for some ten minutes at 600° C. and subsequent slow cooling makes a satisfactorily vacuum-tight joint. The glass to which the mica is to be sealed should be selected to have a coefficient of thermal expansion approximately equal to that for mica. Mica used was 0·005" to 0·02" thick. Heating above 600° C. should be avoided, as the mica will tend to split into laminae.

It is frequently necessary to seal windows of glass on to metal vacuum vessels so as to observe the progress of the operation taking place inside the vacuum. This is done using Picein wax applied hot to the union, both glass and metal having been previously warmed to about 50° C.

Alternatively, a rubber gasket or an "O" ring may be used. The aperture in the metal chamber must then have flat, ground edges ; the rubber ring is placed between this flange and the glass window and a metal clamping ring devised.

A conical, oil-sealed joint is shown in Fig. 47. Other methods

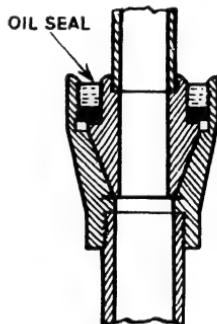


FIG. 47.—An Oil-sealed Union.

¹ J. S. Donal, *ibid.*, 13, 266, 1942.

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of arranging an oil-seal about a join have been undertaken (*see B.P. No. 451,417*).

In some types of experimental vacuum systems it is very useful to be able to move some mechanism or object inside the chamber

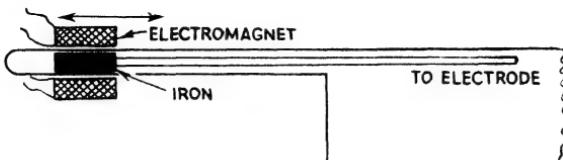


FIG. 48.

without letting down to atmospheric pressure. Some useful ways of doing this are shown in the accompanying figures.

In Fig. 48 is shown a magnetic control for shifting, say, one of the component electrodes of an electron gun assembly. A linear or rotatory motion can be imparted in this way. Fig. 49 shows the use of a rotatable stop-cock which, winding up the attached

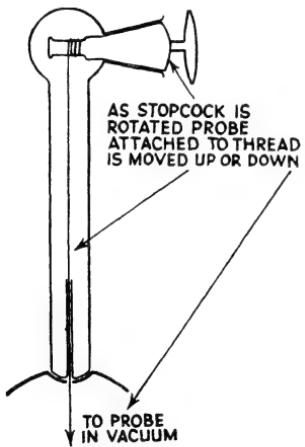


FIG. 49.

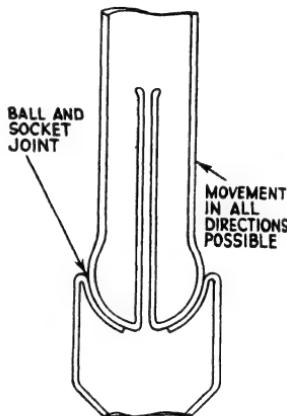


FIG. 50.

thread, allows the target in the vacuum to be raised or lowered. Similarly a rotatable cone joint in metal can be used to operate a flap in vacuum, enabling a small part of the system to be let down to atmospheric pressure for the insertion of a new specimen whilst the main part of the system remains at low pressure. This is

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applicable to the vacuum spectrograph and the electron microscope as a method of facilitating the rapid change of photographic plates. A ball-and-socket type of ground-glass joint enables a more universal motion to be arranged. See Fig. 50.

Another method of moving a probe in vacuum by outside control in the air is shown in Fig. 51, where a flexible brass or tombac

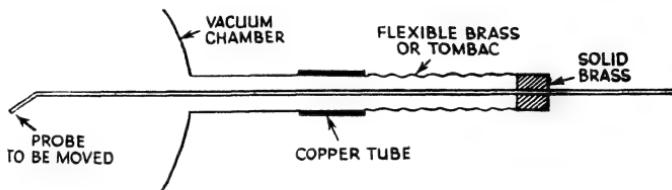


FIG. 51.—Moving a Probe in Vacuum.

tubing allows the rod connected to the probe to be moved in any direction over a small distance.

Delsasso and Creutz describe a quick-acting vacuum lock (Fig. 52) which makes use of rubber or neoprene gaskets to enable a specimen to be rapidly inserted into a chamber without breaking the vacuum.

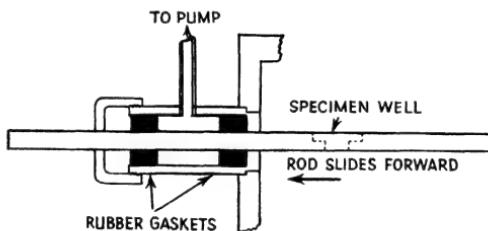


FIG. 52.—Inserting a Specimen into Vacuum.

Vacuum valves, as distinct from stop-cocks but performing much the same function, employ rubber or mercury as the sealing agent in most cases. A typical example is shown in Fig. 53.

A great many types of vacuum valves, rubber-seated and otherwise, have been described in the literature; almost every month some new arrangement is described, usually for some particular application. The Saunders valve,¹ originally introduced for

¹ Saunders valves suitable for high vacuum work are supplied by W. Edwards and Co., Ltd.

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high-pressure work and steam engineering practice, is a reliable, rubber-seated valve which, after extra machining in the case of the

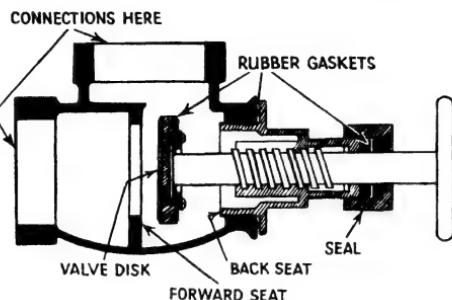


FIG. 53.—A Vacuum Valve.

large sizes, serves very well as an economical cut-off valve for metal vacuum systems. These are available in sizes ranging from $\frac{1}{2}$ " to 3". Again, manufacturers¹ supply specially designed valves

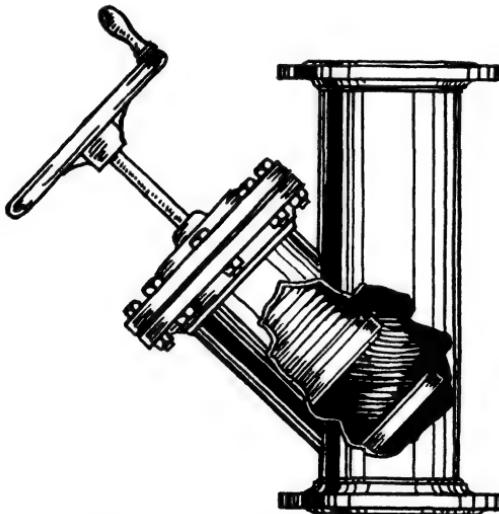


FIG. 54.—A Packless Vacuum Valve.

(Courtesy of Vacuum Industrial Applications Ltd.)

with tube diameters ranging from 1" to 16". Fig. 54 illustrates a 6" pattern.

¹ E.g. Vacuum Industrial Applications Ltd., Wishaw, Scotland.

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An "O" ring rubber gasket is incorporated in the valves used by W. Edwards and Co., Ltd., to cut off their large oil diffusion pumps from the vessel. The metal disc intrinsic in this valve also serves, when open, as a baffle, of which the angle of tilt can be varied, to reduce the amount of back-streaming oil vapour entering the vessel (*see Figs. 30 and 55*).

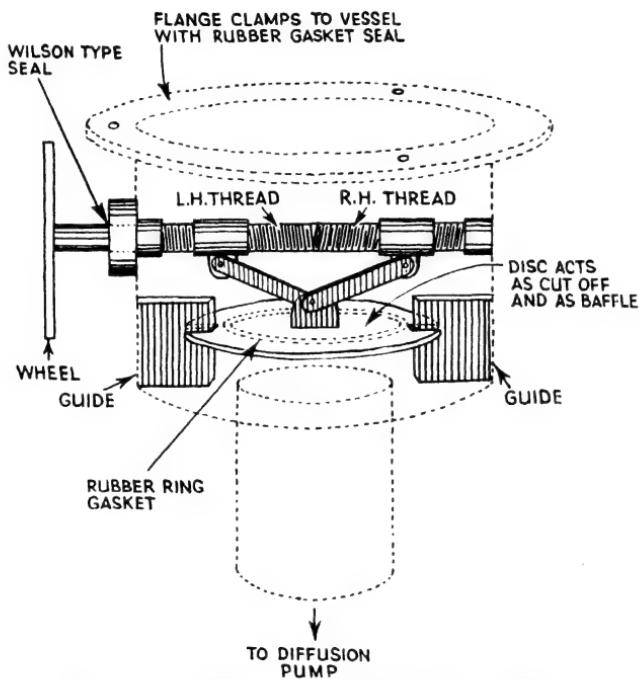


FIG. 55.—Valve for Large Oil Duffusion Pump.

At the other end of the scale, this same firm supplies a small air admittance valve, readily connected in an all-metal system to serve the function of the tap indicated in Fig. 4 (*see Fig. 56*). This valve is often used as an auxiliary to a pot-shaped, metal phosphorus pentoxide trap.

The demand for semi-automatic operation of vacuum plant in mass production techniques has also led to the adoption of electrically and compressed-air operated valves¹ (Fig. 57). Such

¹ Supplied by Metropolitan-Vickers Electrical Co., Ltd., and W. Edwards and Co., Ltd.

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valves can be attached to a number of independent pump units engaged in large-scale routine work, and yet all operated simultaneously by pressing a switch or turning on a compressor. The

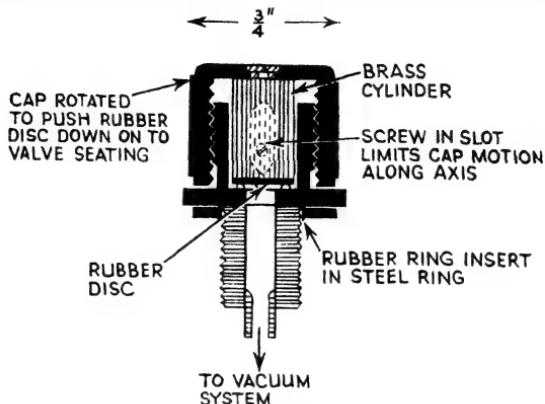


FIG. 56.—Air Admittance Valve.

electrical types are also valuable in providing valves which can be operated via a relay by the output of some form of low-pressure gauge such as the Pirani or thermo-couple gauge (see Chapter Two),

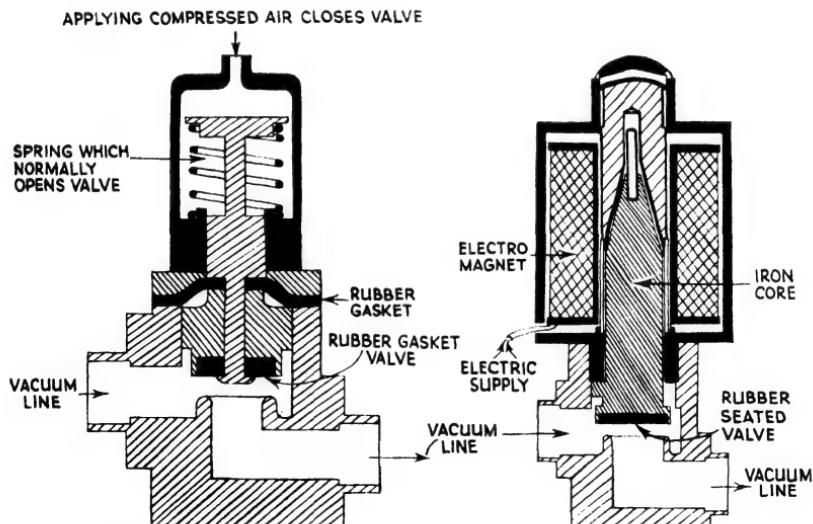


FIG. 57.—Compressed Air and Magnetically Operated Valves.

(Courtesy of W. Edwards and Co., Ltd.

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leading to the automatic operation of a valve at a suitable time in the pump down cycle. Small magnetic valves¹ are also available which are energized by the same electric supply as that to the rotary pump motor. On switching off, the valve automatically shuts off the system and admits air to the rotary pump.

Though rubber-seated valves are very convenient and are widely used in vacuum plant, yet they have two notable disadvantages for low-pressure work. Firstly, the rubber exerts a vapour pressure of the order of 10^{-5} mm. Hg. Secondly, the valve and associated assembly cannot be readily de-gassed by heating (see Chapter

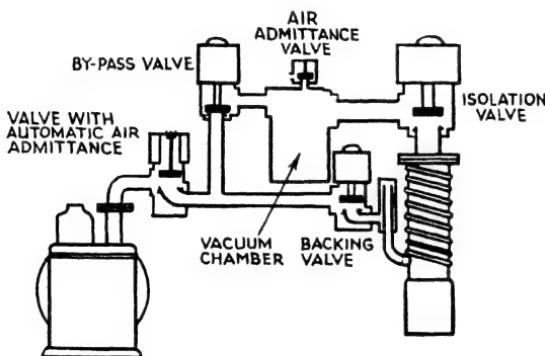


FIG. 58.—Diagrammatic Layout showing Some Applications of Magnetic Valves in an Automatic System.

(Courtesy of W. Edwards and Co., Ltd.)

Four). If the vacuum vessel is shut off from the pumps when a pressure of, say, 10^{-6} mm. Hg is attained, it is impossible to prevent a subsequent pressure rise in a matter of a few minutes, due to gases and vapours evolved from the rubber and the metal walls of the assembly. This is not so important if pumping is continuous but, even so, the ultimate vacuum will be decided by the rate of gas evolution compared with the rate of pumping. A greased glass stop-cock as an alternative cannot be de-gassed by heating either. Moreover, the grease exerts a vapour pressure and also evolves dissolved gases.

In a remarkable series of experiments to produce ultra-high vacua of the order of 10^{-10} mm. Hg and less, and where the leak rate into

¹ W. Edwards and Co., Ltd.

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a glass vessel sealed from the pumps by a valve gave a rate of rise of pressure of only 10^{-12} mm. Hg/sec./litre, Alpert¹ devised an all-metal valve without grease which could be de-gassed at a tem-

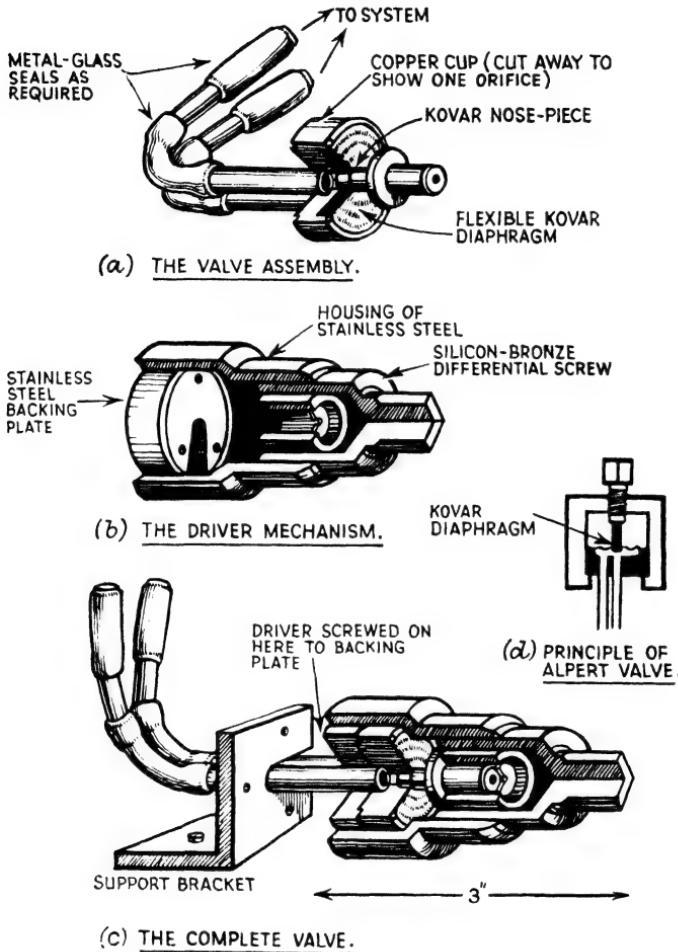


FIG. 59.—Valve for Ultra-high Vacuum Work (Alpert).

perature of 400° C. This valve also provides an excellent "needle valve" for the handling of very pure gases in vacuum systems.

Alpert's valve has a $\frac{1}{4}$ " diameter orifice, but this could be enlarged

¹ D. Alpert, *Rev. Sc. Inst.*, 22, 536, 1951, and *Jour. Appld. Phys.*, 24, 860, 1953.

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in a modified design. When closed, its effective conductance (see Chapter Three) for nitrogen is 10^{-11} litre/sec. or less. If, for example, a one-litre vessel filled with gas were isolated from the pumps by a valve with a conductance of 10^{-10} l/sec., it would take several years for the pressure in the vessel to drop by 1% due to leakage through the valve. Again, at 10^{-9} mm. Hg, there is no significant pressure change recorded on opening or closing the valve.

This unique valve is in two sections : (a) the valve assembly and (b) the driver mechanism (Fig. 59) which opens and closes the valve ; this driver mechanism is removed completely during a high-temperature bake, the valve being meanwhile retained in its open position by a U-shaped stainless steel strap called a "bake-out clamp".

The valve assembly, which is permanently attached to the vacuum system, consists of a copper cup 1.75" diameter with 0.25" diameter holes for the vacuum orifices. A flexible Kovar diaphragm, which allows to and fro of about 0.1", is silver-brazed in a hydrogen atmosphere to the copper cup at its periphery and to the Kovar nose-piece at a central hole. This Kovar nose has a highly polished, 45° conical surface which forms its own seat in the copper cup when the valve is closed for the first time. All the joints are brazed using silver-copper solder, without flux, in a hydrogen furnace. The valve assembly produced is then free of surface oxides and completely clean.

The driver mechanism (Fig. 59b) is separate and removed during baking. To operate the valve when cold it is screwed into position (Fig. 59c). The most satisfactory device consists of a differential screw mechanism with a large mechanical advantage which moves the Kovar nose about 0.01"/rev. and provides a total travel of 0.1". This driver mechanism is in four parts : (i) an outside screw and housing of stainless steel, (ii) a silicon-bronze differential screw with a thread pitch of 20/inch outside and 26/inch inside, (iii) the stainless steel drive screw which is fastened to the valve nose, (iv) a stainless-steel backing plate, securely and rigidly bolted to a bracket on the bench on which the vacuum system is mounted. This backing plate is to withstand the large forces exerted upon the copper cup.

The differential screw has a 0.5" square head which is rotated by a torque wrench. This enables forces of 5 to 10 tons to be exerted

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on the Kovar nose, forcing it against the 0.25" orifice in the copper cup to give a practically perfect vacuum seal.

Due to slight oxidation and scorching of the smooth metallic surfaces and the collection of minute foreign particles on the valve seat, the valve deteriorates somewhat in use. Nevertheless, Alpert asserts that, after a year's use, two valves which had begun with a conductance, when closed, of 10^{-11} l/sec. had a conductance value of only 10^{-10} l/sec. when closed with a torque of 20 lb./ft.

The valves were joined in the glass vacuum system at appropriate points using glass-metal seals. The whole system, including valves but with driver mechanisms removed, could then be de-gassed by baking up to 400° C.

Alpert's work on the production and measurement of ultra-high vacua is considered on pages 95 and 92.

CHAPTER TWO

The Measurement of High Vacua

Gauges for the measurement of low pressures may be operated in a variety of ways, depending on the variation of certain physical properties of the gas as the pressure changes. There are only four or five gauges met with in usual practice.

The Discharge Tube.¹ A good indication of the order of pressure in a system can be obtained by ionizing the residual gas and causing a discharge glow. The best procedure is to seal a discharge tube

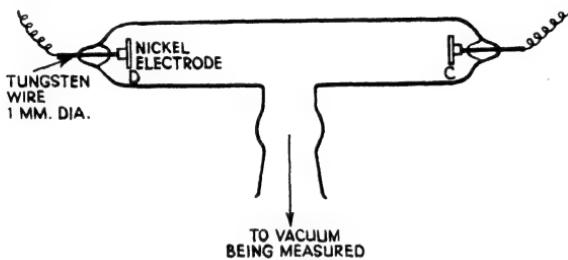


FIG. 60.—The Discharge Tube.

on to a side-tube of the system. This sealing can be done by glass-blowing or by a hard-waxed conical joint (Fig. 60).

The glass bulb is preferably of Pyrex. D and C are two metal plates held inside this tube by means of metal-to-glass seals. These seals also serve as contacts for applying a high potential. The metal plates or electrodes are made of platinum, nickel or stainless steel, and are of fair dimensions so that excessive temperature rise during the discharge is prevented.

The discharge obtained depends on the nature and the pressure of the gas, the current passing and the electrode disposition and size.

At a pressure of from 1 to 20 mm. Hg a streamer of discharge passes from one electrode to the other. At the higher pressures (order of 20 mm.) this streamer is narrow. In the neighbourhood

¹ A very convenient type of shielded discharge tube is supplied by Metropolitan-Vickers Electrical Co., Ltd.

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of a pressure of 1 mm. the streamer widens to the walls of the tube. As the pressure is still further decreased below 1 mm. of mercury definite regions in the glow discharge can be observed. These effects are illustrated in Fig. 61.

Surrounding the cathode there is the cathode glow. This takes up the contour of the cathode surface. Then follows the Crookes dark space, and then the negative glow. From the positive end of the tube extends the positive glow, this is either continuous or striated. The negative and positive glows are separated by the Faraday dark space.

The results give the order of pressure only, i.e. they serve as a preliminary indication of the pressure in the vessel. However,

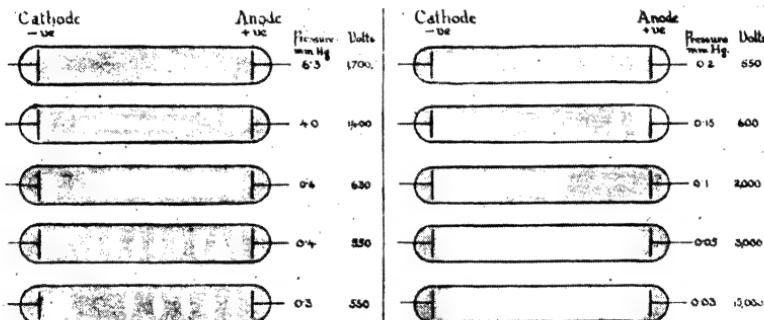


FIG. 61.—Appearance of a Discharge Glow at Various Pressures.

the appearances can be put on some sort of quantitative basis because it has been found that the width of Crookes dark space depends on the mean free path and so on the pressure. The relation can be expressed as

$$p = \frac{k}{L} = \frac{k_1}{d} \quad . \quad . \quad . \quad . \quad (8)$$

where p is the pressure of the residual gas in millimetres of mercury, and d is the width of the Crookes dark space in millimetres. This is a rough relation only, for Aston and Watson¹ have shown that the width also depends on the current density to a small extent.

At pressures lower than 10^{-1} mm. of mercury, the walls of the discharge tube begin to fluoresce. This fluorescence occurs, then, at a good backing pressure, i.e. one attained with mechanical pumps. The pressure at which the fluorescence commences also

¹ Aston and Watson, *Proc. Roy. Soc.*, **86**, 168, 1911.

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depends on the nature and age of the glass, and on the applied voltage. At pressures less than 10^{-2} mm. Hg the discharge disappears if the applied electric field is less than about 100 V per mm. : this state is called a "black" discharge.

The discharge may also be excited by means of currents induced in the gas by a high-frequency oscillator, or by a Tesla coil. The latter method is valuable when a glass vessel is being pumped and the system has no discharge tube. The appearance of the glow indicates when the pressure is a good backing pressure, and that the diffusion pump heaters can be safely switched on. This state is indicated by the glow pervading the whole system, and without any trace of the pink nitrogen discharge : a pressure less than 10^{-1} mm. Hg.

Gas	Cathode glow	Negative glow	Positive glow
Air	Red	Blue to pink	Pink : blue at lower pressures
Ammonia . . .	Blue	Green-yellow	Blue
Argon	Red-pink	Blue	Violet-red
Helium	Red	Pale-green	Violet-red to yellow-pink
Hydrogen	Brown-pink	Light-blue	Reddish-pink
Mercury Vapour	Green	Yellow-white	Greenish
Neon	Yellow	Orange	Blood-red
Nitrogen	Red-pink	Blue	Reddish-yellow
Oxygen	Red	Green-yellow	Lemon-yellow with reddish core
Water Vapour .	White-blue	Blue	White-blue

Variation of the Colour of the Discharge with the Nature of the Gas.

The table shows how the colour of the discharge depends on the gas used. This can be made a scientifically accurate method by examining the discharge with a spectroscope (for the corresponding wave-lengths of the radiation, see Chapter Six). In the case of air the colour of the discharge changes from predominantly pink to chiefly pale-blue as the pressure is decreased. This is because the relative proportions of the gases present in air change as the pressure is lowered, the blue colour at the lower pressures being caused by the increased carbon dioxide content of the air, since this gas has a higher molecular weight, and hence a smaller rate of diffusion to the pump than the nitrogen and oxygen. Moreover, if a mechanical pump only is being used to evacuate the discharge tube, then the

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condensable gases remain at the lower pressures, because they are not withdrawn by such pumps.

The discharge tube method is quite a satisfactory means of indicating the pressure for a great deal of vacuum work. Examples are the systems used for the evaporation of metals *in vacuo*, the fractional distillation of chemicals, the wax impregnation of electrical components, and in the food-canning and dehydrating techniques.

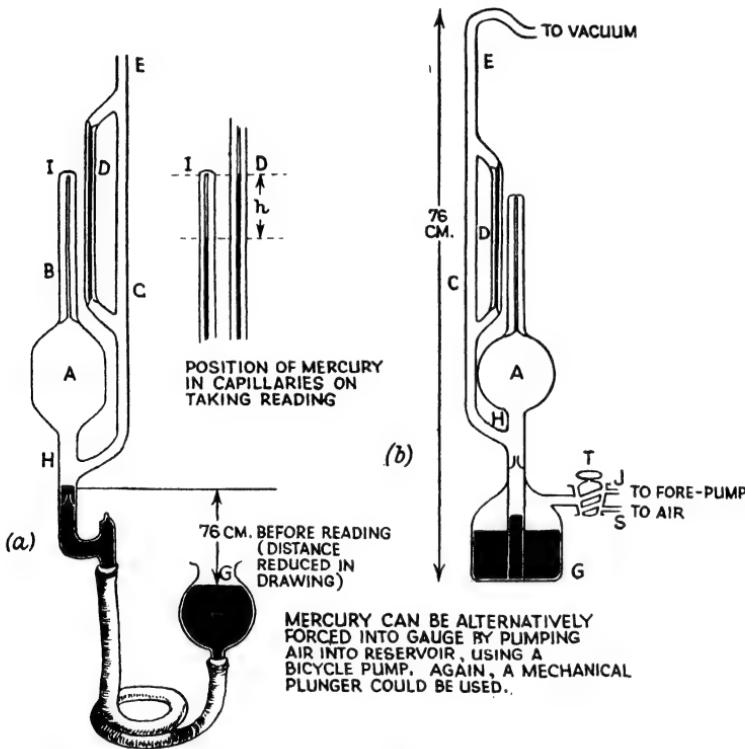


FIG. 62.—(a) McLeod Gauge, (b) Short form of McLeod Gauge.

If a discharge tube of known dimensions is used, and with a certain A.C. or D.C. voltage across it, then when the discharge goes "black" is a satisfactory means of indicating an adequate pressure in many circumstances. Such a procedure as this can be made roughly quantitative by using an adjustable spark gap across the high-voltage supply as an indication of the potential difference applied. The higher the voltage at which a "black" discharge

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is obtained, the lower the pressure will be. Also the discharge method is valuable in that the presence of impurities in the form of condensable vapours is indicated. Thus water vapour and grease vapour from stop-cocks, so often nuisances in vacuum systems which cannot be heated above room temperature, are made evident by characteristic whitish-blue and blue glows respectively. It is worth realizing that the presence of even a small percentage of such impurities often completely spoils the correlation between the nature of the gas and the discharge colour indicated in the table.

The McLeod Gauge.¹ To put the measurement of the vacuum on a good quantitative basis, the McLeod gauge is used. Fig. 62 shows two convenient forms of the gauge.

A glass bulb *A* is attached to a fine capillary tube *B*. *C* is a side-arm provided with a capillary at *D* which has the same bore as the *B* capillary, and runs close to and parallel with it. The vacuum to be measured is connected to the gauge via the tube *E*. The glass system is first thoroughly cleaned and filled with mercury up to the point marked *H* in the figure. In Fig. 62*a* this mercury level can be raised by lifting bulb *G* so that if the gauge is evacuated then it is completely flooded with the mercury. In Fig. 62*b* the top of the gauge is less than 760 mm. above the mercury level in the reservoir, and so the gauge will be normally filled with mercury if it is pumped free of air. To draw it out of the gauge, the mercury is sucked down by pumping the reservoir at *G* through tube *J* with a backing pump. To permit the mercury to rise again, an inlet into the reservoir is provided at *S*. This air-inlet can be turned on or off by means of the tap *T*.

To read the pressure the mercury is allowed to rise. A sample of the gas to be measured is trapped by the rising mercury in the volume between the marks *H* and *I* on the gauge. *H* is the point where the side-arm *C* is joined to the main gauge. Suppose this volume, i.e. the volume of bulb plus capillary, is *V*. This is found by standard procedure before the gauge is set up. The mercury goes on rising until the level in the side-arm *D* is opposite the top end of capillary *B*. Then the rise is stopped, either by bringing the reservoir bulb to rest or by closing the tap *T* of Fig. 62*b*. This mercury rise will compress our sample volume of gas into the capillary so that it occupies it for some length *h*. The cross-section of

¹ McLeod, *Phil. Mag.*, **47**, 110, 1874.

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the capillary is measured before setting up the gauge. Suppose it equals a . Then the gas sample has been compressed from volume V to volume ah , and the head of mercury which is compressing it to this volume is also h . Applying Boyle's law for the perfect gas gives

$$p \cdot V = ah \cdot h \quad . \quad . \quad . \quad . \quad (9)$$

where p is the pressure of the gas which is required, and h is in millimetres of mercury ; therefore

$$p = \frac{ah^2}{V} \quad . \quad . \quad . \quad . \quad (10)$$

a and V are constants in this relation, and so the pressure can be measured in millimetres of mercury on a square law scale.

The comparison capillary being of the same bore as the major capillary eliminates errors due to surface tension.

If the bulb A is about 300 c.c. and the diameter of the capillary tube 1 mm. then a pressure of 10^{-5} mm. Hg can be readily measured, and a 10^{-6} pressure indicated. For a very good vacuum, i.e. one less than 10^{-6} mm. Hg, the mercury will completely fill the capillary, and on allowing the mercury to drop to take a fresh reading, the surface tension will cause it to stick against the closed end of the capillary B until the weight of the falling mercury pulls it away. This is referred to as a "sticking" vacuum.

The McLeod gauge is the most used gauge in vacuum practice, and all the other gauges described below are calibrated against it. Its chief advantage is that it gives, in a straightforward manner, an absolute measurement of the vacuum. It suffers from one great disadvantage : that it will not accurately register the pressure due to condensable gases and vapours. A second, minor trouble is that the gauge must, inevitably, be connected to the system by a fair length of narrow tubing and so it usually takes about one minute for the gauge and system pressures to equalize. (See Chapter Three for the restricting effect of tubing on the flow of gases at low pressures.)

The Vacustat.¹ For measuring pressures in the range 0·01 to 10 mm. Hg, this convenient gauge is a modification of the McLeod which avoids the use of a movable reservoir of mercury, and also requires only about 8 c.c. of mercury for filling.

¹ By courtesy of W. Edwards & Co., Ltd. See E. W. Flosdorff, "A Simplified and Portable McLeod Gauge", *Ind. Eng. Chem.*, **10**, 534, 1938.

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The construction of this gauge is shown by Fig. 63. The overall height is only 9". The vessel containing the gas of which the pressure is required is connected by flexible rubber pressure tubing to the join at *A* on the gauge, so that bodily rotation of the vacustat through a right angle about *A* as centre is possible. Before measuring the pressure the gauge is in the horizontal position (Fig. 63*a*), so that the gas to be measured fills the gauge at the correct pressure. On requiring to take a pressure reading the gauge is simply rotated to the vertical position (Fig. 63*b*), when the mercury runs out of the container *B*, compressing a sample of the gas contained within

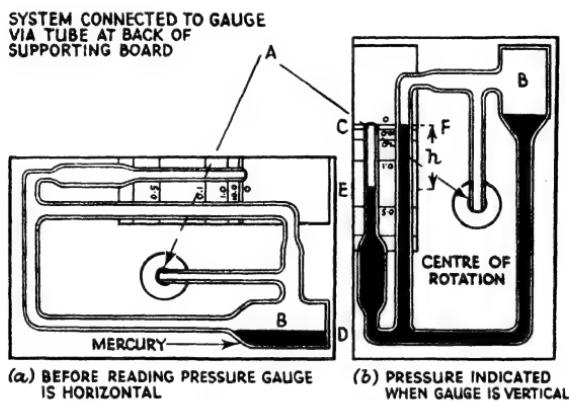


FIG. 63.—The Vacustat (W. Edwards and Co., Ltd.).

the region *CD* to a short length of the capillary tube *CE*. By arranging that the level in the comparison capillary *F* is opposite the end of the graduated capillary *CE*, it is possible to employ a scale behind this capillary which reads the pressure directly. A slight adjustment of the gauge about the vertical position is needed to adjust the mercury level correctly. The method of calibration is the same as for the McLeod gauge, a square law scale being obtained.

The Optical Bellows Manometer. East and Kuhn¹ have described a gauge which is valuable for measuring differences of pressures in the range 10^{-2} to 5 mm. Hg, where the upper limit can be extended to 100 mm. Hg; it is especially useful for measuring pressure

¹ H. G. East and H. Kuhn, *Jour. Sc. Inst.*, 23, 185, 1946.

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differences of round about 1 mm. Hg, which are normally recorded by the ordinary U-shaped manometer, using a low-vapour pressure oil, or mercury. This instrument avoids the difficulties associated with such manometers in addition to being more sensitive. Thus oil gives off absorbed air, may get into the apparatus, and its level is not easily read precisely, whilst the mercury manometer cannot easily be read to an accuracy better than 0.5 mm. Hg.

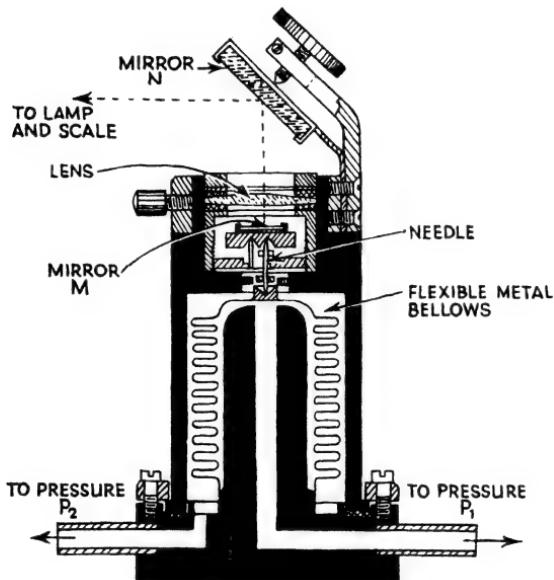


FIG. 64.—The Optical Bellows Manometer.

The gauge is shown in Fig. 64. Flexible bellows of tombac or beryllium-copper separate regions of gas at pressures P_1 and P_2 . When P_1 is different from P_2 , the bellows are displaced, their top moves up or down, depending on which is the greater, P_1 or P_2 . This displacement is transmitted via the vertical, double-pointed needle shown to cause a tilt of a small platform which supports the plane mirror M . A small lamp at some 40 cm. from the gauge projects a beam of light on to the front-aluminized mirror N which is set at 45°. This light beam is thus reflected to the tiltable mirror M . The beam reflected from M will be turned through twice the

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angle of tilt of the mirror. The deflection of the beam is measured by a scale just above the projector lamp, i.e. the usual galvanometer lamp and scale technique is used. The lens shown is fitted into the case with air tight rubber gaskets.

Using a flat scale 40 cm. distant from the manometer, the deflection is linear over a range of 10 cm. Hg, and can be read to 0·1 mm. Hg.

The commercial model¹ of this instrument has the bellows manometer unit, projector lamp, scale, and pressure equalizing and isolation valves housed in a small steel cabinet. Two models are available: model 1 has a single range of 0 to 15 mm. Hg; model 2 is somewhat larger, and employs a striped mirror inside the manometer unit so that the normal reflected beam appears on the scale together with the second- and third-order reflections. This gives three ranges: 0 to 3 mm. Hg; 0 to 0·75 mm. Hg and 0 to 0·5 mm. Hg.

Such a manometer can be used for measuring differential pressures at any mean pressure up to several atmospheres. When used as a high vacuum gauge, however, one side, say P_2 , is connected to a two-stage rotary pump so as to be evacuated to 10^{-3} mm. Hg approx., whilst the other side, P_1 , is linked to the vessel in which the pressure is required.

These gauges read total pressure, since they respond equally well to mixtures of gases and condensable vapours. The calibration is independent of the nature of the gas. The only type of gas which cannot be used is one which attacks copper, such as a halogen.

The Pirani Gauge² is based on the physical principle that the thermal conductivity of a gas is related to its pressure by the approximate relation :

$$K = \alpha \cdot p \quad . \quad . \quad . \quad . \quad (11)$$

for pressure below values where the gas layer thickness is of the order of the mean free path. K is the thermal conductivity of the gas, p its pressure and α is a constant. The thermal conductivity is indirectly measured by ascertaining the change in resistance of a hot filament in the vacuum with pressure. As the pressure round the filament alters, so the speed at which heat is conducted away

¹ Courtesy of W. Edwards and Co., Ltd.

² Pirani, *Verh. d. Deutsch. Phys. Ges.*, **8**, 686, 1906.

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from the filament changes. This effect changes the filament temperature, and hence its resistance. These resistance changes are recorded by a Wheatstone bridge arrangement.

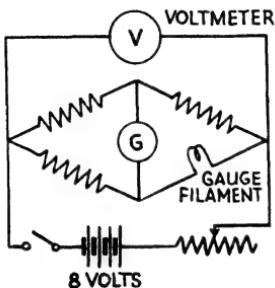


FIG. 65.

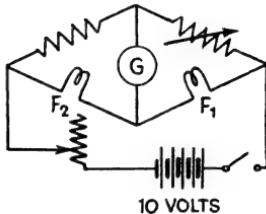


FIG. 66.

Details of the practical set-up used are as follows :

Two methods have been found useful, the circuit diagrams being given in Figs. 65 and 66. These methods are : (1) keep the heating current constant and measure the change in resistance as the pressure is varied ; (2) keep the temperature and so the resistance constant and measure the change in voltage supplied as the pressure is altered.

The best balancing resistance in the bridge arm opposite to the gauge filament is an identical filament sealed off in a bulb at the lowest possible pressure. This compensates for the variations extraneous to the circuit. The filament used should be of a metal with as high a temperature variation of resistance as possible : a tungsten or platinum wire of diameter about 0.03 mm. is recommended. This filament should be suspended on glass beads around a central glass supporting rod, and arranged so that the outer wires are equally spaced from the glass envelope. Fig. 67 shows a suitable arrangement. A standard 40-watt lamp bulb also serves fairly well. If very accurate readings are desired, then the compensating bulb should be thermostated at freezing point. In both types of circuit used, the bridge galvanometer should have a

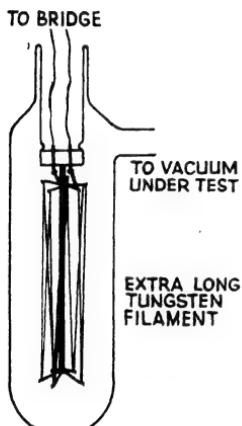


FIG. 67.—A Pirani Gauge.

the outer wires are equally spaced from the glass envelope. Fig. 67 shows a suitable arrangement. A standard 40-watt lamp bulb also serves fairly well. If very accurate readings are desired, then the compensating bulb should be thermostated at freezing point. In both types of circuit used, the bridge galvanometer should have a

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sensitivity of the order of 10^{-8} amps. per division. The instrument has to be calibrated against a McLeod gauge.

Using the second method, due to N. R. Campbell,¹ if V is the potential for a pressure p in the gauge, and V_0 is the potential for the lowest possible pressure, then

$$\frac{V^2 - V_0^2}{V_0^2} = k.p \quad . \quad . \quad . \quad (12)$$

is an approximate relation. Fig. 68 shows the calibration of a typical Pirani gauge using this method.

The gauge is sensitive to vibration owing to the fragile nature of the filament. The bulbs containing the filaments are best mounted in some form of shock absorber.

The total pressure is recorded by the gauge, i.e. it records the pressures of condensable gases and vapours, e.g. water vapour. Unfortunately the heat loss of the filament is determined by the nature of the gas molecule and so the gauge can only really be accurately calibrated for one particular gas. It is very important, therefore, to avoid the introduction of excessive water vapour when measuring air pressures. Despite this fault it has, however, the virtue that it will indicate the presence of unsuspected vapours which the McLeod gauge would completely fail to record. The other advantage is the ability to give the pressure instantaneously.

The useful range of the Pirani gauge is from 1.0 mm. Hg to 10^{-4} mm. Hg.

Dumond and Pickels² developed a Pirani gauge with the intention of extending the lowest range of pressures recordable as far as practically possible. They employed a pair of Pirani gauges each of which contained two filaments of about 3 metres of tungsten wire, 10^{-3} cm. in diameter, wound into helices, each helix being

¹ Campbell, *Proc. Phys. Soc.*, 33, 287, 1921.

² J. W. M. Dumond and W. M. Pickels, *Rev. Sc. Inst.*, 6, 362, 1935.

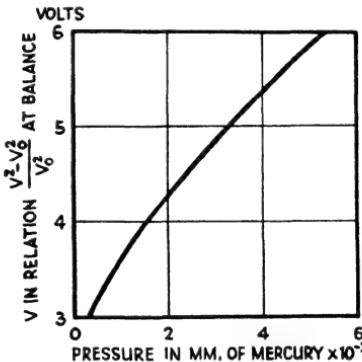


FIG. 68.—Pirani Gauge Curve for Dry Air (using 220 v., 40 w. lamp).

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about 20 cm. long. The four filaments in the two gauges were as nearly equal as possible, and were connected up to form the four arms of a Wheatstone bridge, with a milliammeter to record the off-balance effect produced by a change of pressure in one of the gauges. In this way pressures as low as 10^{-5} mm. Hg were recorded, but it was found that the effect of external temperature and mechanical variations produced an off-balance effect comparable in size with the effect produced by a pressure change of 10^{-5} mm. Hg. In short, the sensitivity of this type of gauge is limited by such considerations, and there is no point in attempting to improve the electrical performance of the circuit further.

Scott¹ arranged a Pirani gauge to apply a potential across the grid to filament of a triode valve, as in Fig. 69. The variations of

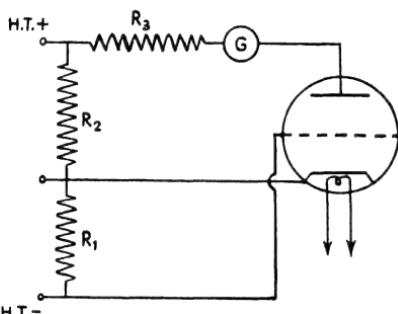


FIG. 69.—Pirani Gauge Circuit (Scott).

voltage across the gauge filament are recorded in terms of the variations in anode current of the valve. The Pirani filament resistance R_1 is about 700 ohms, the resistance R_2 consists of 20,000 ohms fixed resistance in series with a 2000-ohm variable resistance. The resistances R_1 and R_2 form a potential divider across which the H.T. supply is connected. R_3 is an anode load resistance of about 10,000 ohms using a 6C5 type valve, the purpose of which is to give the triode a more nearly linear characteristic for values of grid potential near the cut-off value. As the Pirani resistance R_1 varies with the variation in the conductivity of the surrounding gas in the gauge, so the voltage drop across it varies, altering the grid potential of the valve, and therefore the anode current recorded by the meter G . The total voltage across R_1 and R_2 is constant, being that of the H.T. supply to the valve plus the series anode load, so that when the voltage across R_1 varies with the pressure in the gauge, the voltage across R_2 varies in the opposite direction, i.e. a grid voltage negative increase is accompanied by an anode voltage positive

¹ E. J. Scott, *ibid.*, **10**, 349, 1939.

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decrease, both decreasing the anode current. The meter used in the anode circuit should have a full-scale deflection of about 3 mA.

A commercial type of Pirani gauge¹ which is basically similar to that designed by Dumond and Pickels is illustrated in Fig. 70. A valuable feature of the design is that the maximum possible pressure recordable is extended to as high as 10 mm. Hg.

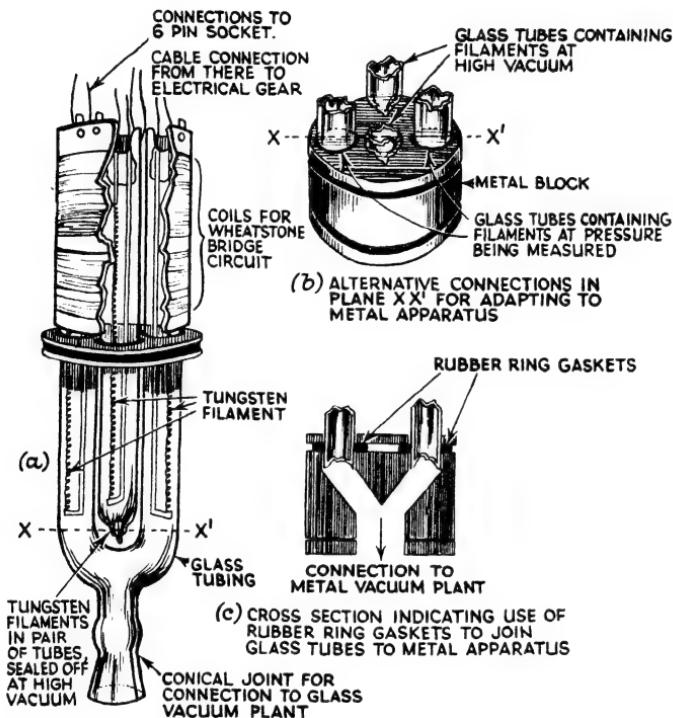


FIG. 70.—Commercial Pirani Gauge Head with Incorporated Bridge Balancing Coils.

The necessary Wheatstone bridge circuit balancing coils are here included on a former placed around the gauge head, which is of the four filament type. The complete assembly is then housed in a robust plastic or metal cylindrical container. If connexion to a glass vacuum system is desired, the type shown in Fig. 70a is used, which is readily attached by a greased cone joint. For use with

¹ Courtesy of W. Edwards and Co., Ltd.

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metal vacuum systems, type Fig. 70b is preferable, where the two Pirani glass tubes which need to be joined to the system are mounted by small air-tight rubber "O"-rings in a metal block. A multi-wire cable joins the gauge head to the mains or battery-operated unit which supplies the necessary electrical power, incorporates a two-range meter directly calibrated in pressure units for dry air, and is furnished with calibrating and filament reconditioning controls. By changing gauge heads, the pressure ranges can be : (1) 10 mm. to 0·5 mm. Hg, and 0·5 mm. to 0·005 mm. Hg, using an atmospherically balanced bridge, i.e. the two compensating filaments are sealed off in tubes at atmospheric pressure ; or (2) 1·0 mm. to 0·005 mm. Hg and 0·005 mm. to 0·0001 mm. Hg, employing a vacuum balanced bridge, i.e. the compensating filaments are sealed off in tubes at the lowest possible pressure. Other models are also available. A platinum wire filament is useful for measuring pressures due to chemically active gases. The effective electrical output from a Pirani gauge can be usefully connected to a recorder unit, which continuously and automatically plots on graph paper the variations of the pressure in the system with time. Such automatic pressure recording is especially useful on large-scale vacuum drying plants. Again the electrical output can be linked by a relay to a magnetically operated valve (see Fig. 57), to cause closing or opening of this valve at a prearranged pressure.

Combinations of the McLeod and Pirani Gauges. Pfund suggested sealing a minute Pirani filament in the top of the McLeod capillary. Thus the gas compressed in a certain ratio by the McLeod is measured on the thermal conductivity gauge. This is claimed to record pressures as low as 10^{-7} mm. Hg. However, the practical setting up and reliable operation of such a gauge is difficult because of the small size of the Pirani filament, and so this gauge is little used. Again, the mercury vapour pressure of 10^{-3} mm. Hg will be always present in the closed capillary. To avoid this, it should be possible to devise a metal chamber with an air-tight plunger to compress the gas in a known ratio before its pressure is recorded by a Pirani gauge or an aneroid capsule manometer.

Ionization Gauges. The electrons from a heated filament in a vacuum bulb are accelerated towards a collecting electrode, and the positive ions formed during their passage are collected on a third electrode, and the current recorded varies with the pressure

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prevailing in the bulb. A normal triode valve such as the LS5 may be used for this purpose, but it is better to use a specially constructed electrode system like those illustrated in Figs. 71 and 72 to avoid electric leaks between electrodes.

The filament *F* is heated by the passage of an electric current. This causes electron emission into the surrounding vacuum. *G* is a concentric grid, and *A* the outer concentric anode. The electrons are accelerated towards a positively charged electrode. In one arrangement this is the grid. Some of the electrons pass through the grid spaces and ionize

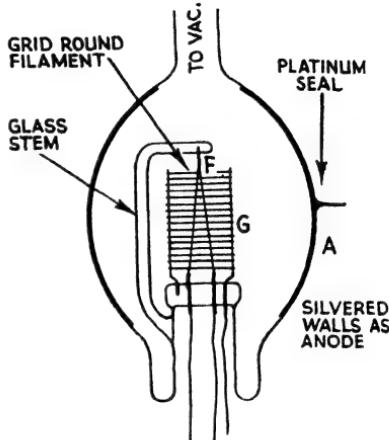


FIG. 71.

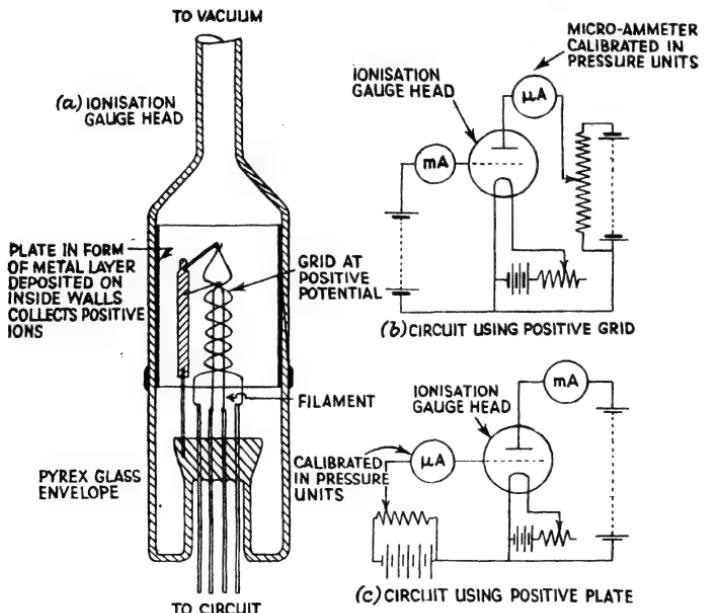


FIG. 72.—The Triode Type of Ionization Gauge.

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the residual gas between the grid and the anode. The anode is at a negative potential so that it collects the positive ions formed. The circuit arrangement is shown in Fig. 72b.

A second scheme is to operate the outer anode at the positive electron-collecting potential and then the grid is made negative so that the grid circuit records the ionization; see Fig. 72c.¹

The negative anode arrangement is the most sensitive method, but the negative grid procedure is easier. The electron current is, in both circuits, measured by a milliammeter, whilst the ionization current is measured by a sensitive microammeter of the lamp and scale pattern using a suspended coil with an attached mirror. The order of the electron current or emission is 10 mA. whereas the ionization current is about 20 microamps at a pressure of 10^{-3} mm. The emitting filament is preferably of tungsten wire without the usual oxide coating. Two filaments are often mounted so that a spare one can be used if one filament burns out or is ruined by letting in air when it is hot. Alternatively a platinum filament may be used, this will not be spoiled by heating at atmospheric pressure, and also the higher range of pressures recordable can be extended, providing an accurate calibration curve is plotted. The grids are of molybdenum or tungsten wire and the outer anode of nickel. It is very important to thoroughly out-gas the gauge at the lowest possible pressure before use. This is done by first baking the containing bulb in an oven at 500° C. and then out-gassing the electrodes either by eddy-current heating or by electron bombardment. (See Chapter Four for details of procedure.)

One ingenious form of gauge utilizes spirals of tungsten wire for both the grid and the anode, so that the out-gassing can be done by heating due to the passage of electric current through the spirals. Getters (see Chapter Four) must not be used because they introduce electric leaks, and also their effect is ruined if the gauge is ever opened to the atmosphere. Every time the gauge is opened to atmospheric pressure the de-gassing must be repeated, and so, as far as possible, the gauge should always be maintained at a pressure below 10^{-2} mm. Hg.

Fig. 73 shows a calibration curve for an ionization gauge.² Below

¹ See Dushman and Found, *Phys. Rev.*, **17**, 7, 1921, also *Phys. Rev.*, **23**, 734, 1924.

² Dushman and Found.

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10^{-4} mm. Hg the pressure is directly proportional to the ionization current. If the emission from the filament is reduced below 5 mA., then the relation is linear up to a pressure of 10^{-3} mm. Hg, but then, of course, the ionization current is smaller. With emissions of 10 mA. approx. the collector voltage needs to be about -20 V. and the accelerator potential of the order of 200 V. If the emission of electrons from the filament exceeds 10 mA., then spurious effects due to space charge upset the linear relation between ion current and pressure.

For the same pressure, anode voltage and anode current, the ionization current is proportional to the number of electrons in the

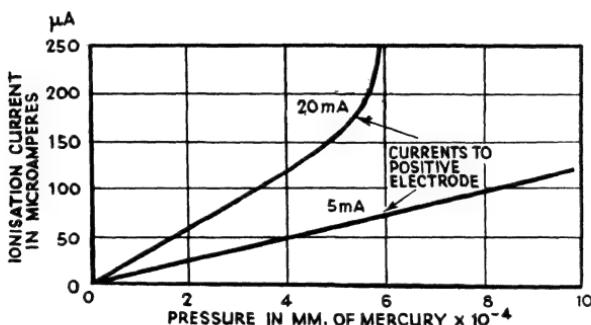


FIG. 73.—Calibration Curve for Ionization Gauge.

gas molecule. Thus the gauge is selective, and needs to be calibrated for each gas used.

The lowest pressures attained have been measured with ionization manometers. Pressures as low as 10^{-10} mm. Hg have been recorded. This extremely low limit to the pressure recordings is the great advantage of the gauge. It is also instantaneous in its action and is, therefore, much used to record pressure variations in electron tubes undergoing pumping and activation procedures.

The two greatest disadvantages of the gauge are its low maximum pressure reading, and the fact that it needs to be calibrated for each gas used.

The calibration is done against the McLeod gauge. Great care needs to be taken to ensure gas purity and equality of pressure throughout the system during this calibration. Readings below

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10^{-4} mm. Hg are interpolated on to the graph since it is known that in this region the pressure-current relation is linear.

R. S. Morse and R. M. Bowie¹ have described a new type of ionization gauge of which the construction is shown in Fig. 72a.

Pyrex glass is used, and the collecting electrode is in the form of a thin translucent film deposited over a portion of the inner wall of the bulb. Electrical contact is made to this platinum film by means of a wire embedded in the glass. External contact is in the form of a metal ring round the centre of the gauge. The advantage of this construction is that the tube can be readily out-gassed by heating with a blow-pipe flame since there is no danger of cracking the seals as used in the older constructions. The grid is a spiral of tungsten wire, arranged so that it can be out-gassed by the passage of an electric current from a 6-V. source applied to the appropriate pinch leads. A pure tungsten wire filament was considered to be most suitable since the electron emission is then particularly stable after long usage.

Increased sensitivity of this gauge is claimed to be attributable to the relatively large volume between the grid and the plate in which ionization occurs, and to the open nature of the grid. The best operating potentials are specified to be 150 V. on the grid with -25 V. on the anode, with an anode current of 5.0 mA. for a filament consuming 10 watts approximately.

Later,² Bowie described a suitable amplifier to enable the small ionization current to be magnified so that it could be recorded on a robust type of meter. The amplifier used was stabilized by using a twin triode balanced circuit, negative feed-back, and gas discharge tube regulation of the voltage supply.

L. N. Ridenour³ describes an ionization gauge circuit in which an electron-ray or "magic eye" tuning indicator valve such as the 6E5 is used to record the ionization current instead of using a sensitive microammeter. The ion collector current of the gauge valve flows through either a 20,000-ohm or a 2-megohm resistance, depending on the position of a switch which alters the range of the instrument. The potential difference across this resistance causes the shadow angle of the 6E5 "magic eye" valve to widen. A poten-

¹ R. S. Morse and R. M. Bowie, *Rev. Sc. Inst.*, **11**, 91, 1940.

² R. M. Bowie, *ibid.*, 265, 1940.

³ L. N. Ridenour, *ibid.*, **12**, 134, 1941.

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tiometer is then used to apply an opposing voltage to this 6E5 so as to bring the shadow angle back to its original setting. This potentiometer can then be calibrated in terms of the size of the gauge ion current, and so of the pressure of gas in the gauge.

Commercial ionization gauge units are available from a number of manufacturers. A type operating from an A.C. supply at 230 V. which incorporates a bridge D.C. amplifier¹ has a useful pressure range from 10^{-2} to 10^{-7} mm. Hg. In this unit, an emission regulator keeps the electron current constant, a stabilovolt is used for the electrode potentials, and a D.C. amplifier measures the positive ion

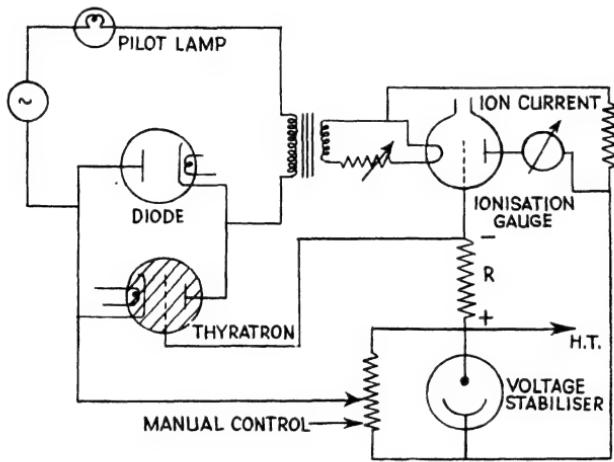


FIG. 74.—Ionization Gauge Control Unit Circuit.

current to the anode, which gives out-of-balance currents of the order of 1 mA. for the positive ion currents obtained. To prevent damage to the gauge should the pressure in it become excessive, a vacuum relay is incorporated which switches off the gauge filament when the pressure is above 10^{-2} mm. Hg.

W. Steckelmacher² describes an ionization gauge control unit employed commercially (see Fig. 74). In this circuit a thyatron is used to modulate the filament current in opposition to fluctuations in emission. The positive grid procedure is adopted. A

¹ By courtesy of Metropolitan-Vickers Electrical Co., Ltd.

² W. Steckelmacher, article on "High Vacuum Gauges" in *Electronics and Research*, ed. A. G. Peacock, Chapman and Hall Ltd., 1949.

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resistance R carrying the electron current to this grid will have a P.D. across it which fluctuates in accordance with variations of emission. This fluctuating P.D. supplies negative bias to the thyratron, which is thus quiescent if the emission, and so bias, is high, and fires if the emission is low. The diode valve included is necessary to ensure a closed transformer primary circuit during the time when the thyratron anode is negative. The filament current is therefore increased or decreased depending on electron emission fall or rise, so that the emission current is maintained within narrow limits of variation.

The Extension of the Low Pressure Range of the Ionization Gauge.¹

It has already been asserted that the ion current for a given electron emission is directly proportional to the pressure below 10^{-3} mm. Hg. That this is true down to 3×10^{-6} mm. Hg was verified experimentally by Dushman.² Assuming that such linearity persists to much lower orders of pressure, indications of vacua at 10^{-8} mm. Hg have been obtained. However, though it is suspected that even lower pressures than this figure are attainable, yet the conventional ionization gauge does not record them because of a constant, residual ion current irrespective of pressure at pressures less than 10^{-8} mm. Nottingham³ suggested that this current was due to soft X-rays. Thus the electrons arriving at the positive electrode with energies of about 150 eV. will produce X-rays with a minimum wave-length of about 100 Å. These X-rays irradiate the negative ion collector and release photo-electrons from its material. These electrons leaving the ion collector form a residual current which is added to the current due to positive ions arriving, and the photoelectric current will be constant irrespective of gas pressure.

To reduce this photoelectric current to as low a level as possible, and hence extend the range of the ionization gauge to 10^{-10} mm. Hg or even less, Bayard and Alpert adopted the simple but ingenious expedient of devising a gauge which is inverted from the conventional arrangement and in which the geometrical cross-section of the ion collector to radiation from the grid is some one hundred times smaller than in a conventional gauge (Fig. 75).

¹ R. T. Bayard and D. Alpert, *Rev. Sc. Inst.*, **21**, 571, 1950.

² S. Dushman, *Phys. Rev.*, **17**, 7, 1921.

³ W. B. Nottingham, see S. Dushman, *Scientific Foundations of Vacuum Technique*, p. 359, Chapman and Hall, Ltd. 1949.

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The filament *FF* is outside the cylindrical grid *G*; this grid acts at a positive potential to collect the thermionically emitted electrons, whereas the ion collector *C* is at a negative potential and consists of a fine wire suspended centrally within the grid. The volume enclosed by the grid is made comparable with that encountered between the grid and plate of the conventional gauge so that a similar volume of ionized gas is concerned. The small interception of X-rays from the grid by the thin wire ion collector results in a greatly reduced production of photo-electrons. The residual constant current due to this effect is thus only about 1% of the value encountered in conventional ionization gauges. Correspondingly, a decrease of the true, pressure-dependent, ion current can be recorded down to pressure of 10^{-10} mm. Hg or even less.

The Ionization Gauge as a Pump. If a vessel with an attached ionization gauge which has been thoroughly out-gassed is pumped down to the lowest possible pressure using, say, a mercury diffusion pump-rotary pump combination with liquid air traps, and the vessel is then sealed off from the pump, it is found that, when the ionization gauge is operated, the pressure in the isolated vessel decreases still further. On switching off the gauge, the pressure rises again.

An ionization gauge therefore has a pumping action. This clean-up of gases is due to two distinct phenomena, (*a*) chemical, (*b*) electrical. The former occurs with the gauge filament on, but without accelerating potentials applied to the other electrodes. Thus a tungsten filament at about $2,100^{\circ}$ C. will react chemically with residual gases and reduce the pressure in the vessel. For example, as pointed out by Alpert,¹ molecular hydrogen dissociates

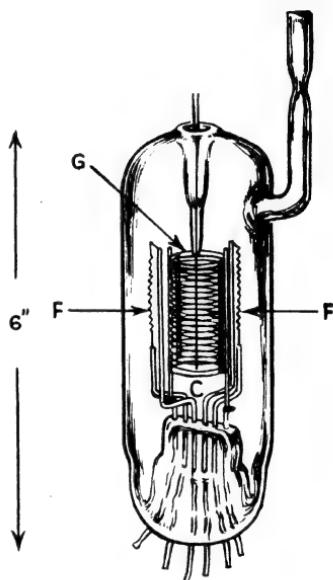


FIG. 75.—Ionization Gauge for Recording Pressures Down to 10^{-10} mm. Hg (Bayard and Alpert).

¹ D. Alpert, *Jour. Appld. Phys.*, **24**, 860, 1953.

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on striking a tungsten filament at a temperature above 1500° C. to form atomic hydrogen. This is very active chemically and is immediately removed from the enclosure on striking and becoming trapped by the vessel walls. Active oxygen, chlorine and some hydrocarbon vapours may be similarly removed. Electrical clean-up of gases (*b*) occurs only when the gauge accelerating potentials are applied. Schwarz¹ shows that this is due to the collection of positive ions at the negative collector. Thus each time a positively charged gas atom or molecule arrives at the collector it gives up its positive charge and the molecule combines with the metal of the electrode. Von Engel² indicates that the speed of an *ion pump* operating by virtue of electrical clean-up alone can be estimated in the following manner.

Assume that every positive ion which reaches the collector gives up its charge and that the molecule concerned is trapped in the metal. If the positive ion current is I_p and each ion has a charge of e , then the rate of collection of ions and so gas molecules is

$$\frac{I_p}{e} = - \frac{dn}{dt} \quad \quad (13)$$

where n is the number of molecules at time t in the enclosure of volume V .

$$\text{Now} \quad p = NkT \quad \quad (14)$$

where p is the gas pressure, N the number of molecules per cubic centimetre, k is Boltzmann's constant and T the absolute temperature. From equations (13) and (14) it follows that the rate of reduction of pressure is given by :

$$\frac{dp}{dt} = kT \frac{dN}{dt} = kT \frac{d(n/V)}{dt} = - \frac{kTI}{Ve} \quad \quad (15)$$

For example, at $T = 300^\circ$ K., $V = 1$ litre = 10^3 c.c., suppose the ion current $I_p = 10 \mu\text{A} = 10^{-5}$ A., then substitution in (15) gives

$$\frac{dp}{dt} = - \frac{1.38 \times 10^{-16} \times 300}{10^3} \times \frac{10^{-5}}{1.6 \times 10^{-19}} \text{dynes/sq. cm./sec.}$$

because $k = 1.38 \times 10^{-16}$ erg./° K, and $e = 1.6 \times 10^{-19}$ coulombs, the charge on an electron, which is equal to the charge on a singly ionized positive ion.

¹ H. Schwarz, *Z. Physik*, **117**, 23, 1940.

² A. von Engel, *Vacuum*, **1**, 257, 1951.

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Converting from dynes/sq. cm. to mm. Hg, where

$$1 \text{ dyne/sq. cm.} = 0.75 \times 10^{-3} \text{ mm. Hg}$$

$$\therefore \frac{dp}{dt} = \frac{-1.38 \times 0.75 \times 3}{1.6} \times 10^{-6} = 1.95 \times 10^{-6} \text{ mm. Hg/sec.}$$

Thus the ionization gauge acting as a pump would be expected to reduce the pressure in a one-litre vessel at the rate of 2×10^{-6} mm. Hg/sec. if the ion current were $10 \mu\text{A}$. This is found to be of the order of magnitude encountered in practice.

The accumulation of gas in the metal of the ion collector will ultimately result in a back-pressure due to diffusion of ions from this electrode back into the chamber. Alpert points out that an ionization gauge operating at 10^{-4} mm. Hg would collect a monomolecular layer of ions (approximately 10^{15} molecules/sq. cm.) on the surface of its collector in one hour approximately. The gauge as a pump would then become saturated and cease to be effective. This conclusion was verified experimentally by Alpert. He asserts, in addition, however, that at a pressure of 10^{-9} mm. Hg, the gauge would be able to pump for 10^8 sec., or three years, before its metal collector was saturated by a monomolecular layer of gas.

The pumping action of the ionization gauge is liable to lead to inaccurate pressure indications if it is overlooked. The development of this phenomenon is, on the other hand, leading to the design of special gauges for pumping purposes at low pressures. The application of such ion pumps to the production of extremely low pressures has been developed particularly by Alpert and his colleagues at the Westinghouse Research Laboratories, East Pittsburgh, Pennsylvania.

Alpert shows that, at a pressure in the range between 10^{-7} and 10^{-8} mm. Hg an oil diffusion pump becomes a source of contamination of a clean vacuum system rather than a pump because of the vapour pressure of its oil. He therefore built a system comprising two Bayard-Alpert ionization gauges (page 92) and Alpert vacuum valves (page 70). The entire system was pumped by a two-stage, glass oil diffusion pump using Octoil S and was baked out at 420°C . The ionization gauges were then thoroughly de-gassed. One gauge was then left running in contact with the diffusion pump whilst the other was isolated by the valve. At first, the pressure in the gauge adjacent to the diffusion pump decreased, because of ion pump

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action, but eventually rose to an equilibrium value of about 10^{-7} mm. Hg, characteristic of the vapour from the diffusion pump oil. The gauge which was sealed off from the diffusion pump continued, on the other hand, to operate as an ion pump for the 900 hours of the experiment, a vacuum of 2×10^{-10} mm. Hg being attained (see frontispiece photograph).

The Philips Ionization Manometer.¹ The pressure is read in terms of the ionization current produced by applying a high voltage across

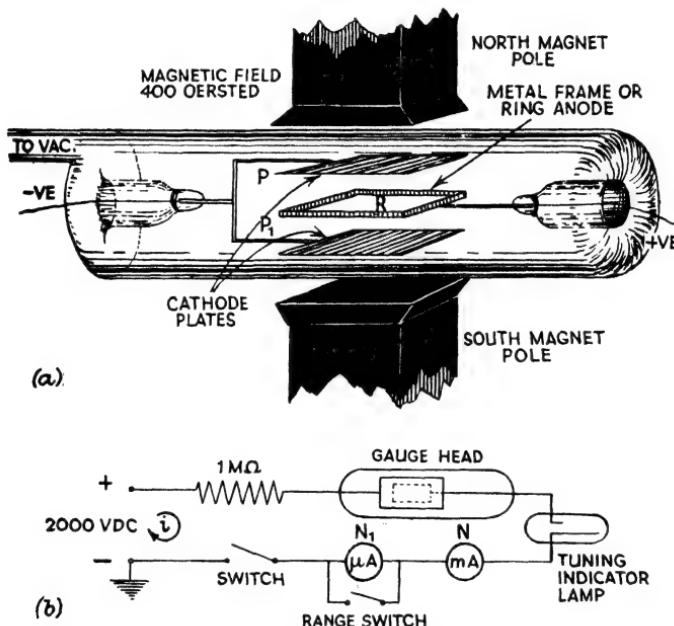


FIG. 76.—The Philips Ionization Manometer.

electrodes sealed into a discharge tube. Electrons emitted from the cathode ionize the gas in passing to the anode. Their path, from cathode to anode, is made several hundred times longer than the direct path by arranging a magnetic field across the tube so that the electrons move in a spiral. Increasing the path of the electron will, in proportion, increase the probability of an electron ionizing the molecules of the residual gas, and so a magnified ionization current is obtained several times greater than that which would

¹ Penning, *Philips' Technical Review*, July, 1937.

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prevail if the electrons travelled in straight lines from cathode to anode impelled by the electric field only. A calibration curve (see Fig. 77) is drawn of ionization current against pressure. Pressures as low as 10^{-5} mm. Hg are instantaneously recorded.

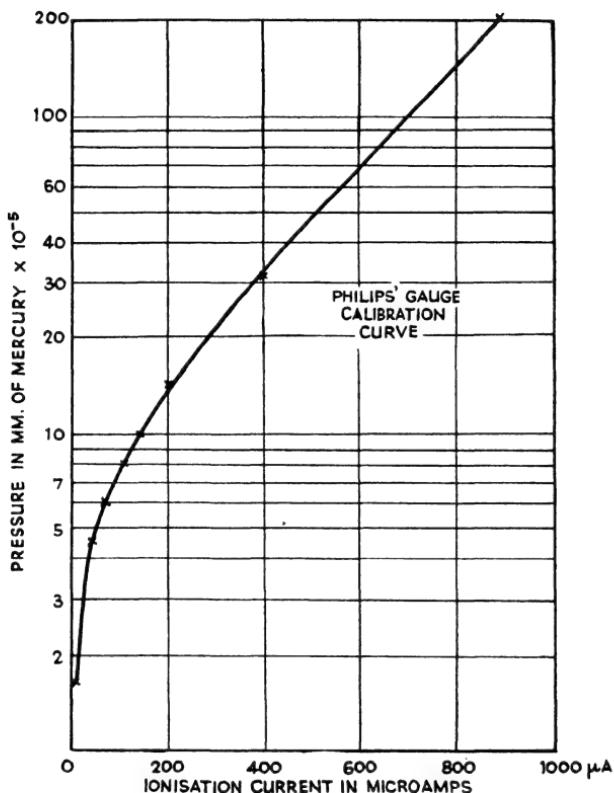


FIG. 77.

The figure 76*a* represents the electrode arrangement. PP_1 is a double-plate cathode, and R a ring-shaped anode. A transverse magnetic field is produced by the permanent magnet. The voltage across the tube should be about 2000 V. D.C. and the magnetizing field should have a strength of some 400 oersted. The manometer is arranged in an electrical circuit as shown in Fig. 76*b*. The supply is conveniently a voltage doubler valve rectifier unit working from

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A.C. mains, the output being smoothed by condensers and chokes, to give 2000 V. D.C. at 20 mA. The ionization current is read by one or other of the meters N , N_1 , N being a milliammeter, and N_1 a microammeter in series. N_1 is shorted by the switch S for large current readings at the higher pressures. Alternatively a tuning glow lamp, such as is used in a radio receiver, will serve as a visual guide to the pressure. The cathode is preferably of zirconium or thorium, giving a copious supply of electrons under the "cold" discharge and less liable to give metal deposits caused by sputtering. The gauge should be re-calibrated for each gas used.

An outline of the theory of the spiral motion of the electrons is given to serve as a guide in the construction of the gauge.

If e = the charge on the electron in electromagnetic units

m = the mass of the electron in grammes

V = the potential between cathode and anode, in electromagnetic units.

then

$$\frac{1}{2}mv^2 = Ve \quad \quad (16)$$

by equating the kinetic energy of the moving electron at the anode to the potential energy at the cathode.

The transverse component of the magnetic field H^* will produce a force F at right angles to the direction of the lines of electric force where F is obtained from

$$F = Hev \quad \quad (17)$$

These two forces acting at right angles will result in the electron moving forwards from cathode to anode with a spiral motion where the radius of the circular component of the motion is given by

$$F = \frac{mv^2}{r} \quad \quad (18)$$

$$\therefore Hev = \frac{mv^2}{r} \text{ or } r = \frac{mv}{He} \text{ where } r \text{ is the radius of the circle.}$$

The theory here given is far from complete, as it does not take into account the variations of mass of the moving charge with pressure. At the higher pressures these negative ions which go to the anode will be ionized atoms, whereas at the lower pressures they will be free electrons. The path will always be helical, however, and so

* Note that there is a component of the magnetic field at right angles to the electric field lines since the cathodes are smaller than the ring anode.

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the total ionization current will be much greater than if the ions travelled straight from the cathode to the anode. Again, the path is increased by the oscillation of the negative ions to and fro about the ring-shaped anode. Thus, the ions, on arrival at the anode plane, will tend to pass beyond it, be drawn back again, and fluctuate about the ring before being finally collected by it.

The Alphatron Gauge.¹ A type of ionization gauge, first developed by Downing and Mellen,² in which the alpha particles from 0.2 milligram of radium, in equilibrium with its disintegration products, ionize the residual gas in a small chamber sealed to the vacuum system. The radium is sealed in a small container to prevent the radon gas formed from being evolved. The ionization current is recorded between a pair of grids across which is maintained a D.C. potential difference of 30 to 40 V. A disadvantage of the gauge is that this ion current is very small, about 2×10^{-11} amp. at 10⁻¹ mm. Hg, so that a D.C. amplifier is practically inevitable. On the other hand, the calibration is linear over a wide range from 10⁻³ mm. to 100 mm. Hg, and there is no filament to burn out. The presence of radium demands that caution be exercised in handling the gauge.

The Thermocouple Gauge. An alternative to the Pirani gauge, in which use is made of the fact that at low pressures the heat conductivity of a gas is proportional to the gas pressure, is the thermocouple gauge. A thermo-junction of thin (approx. 0.05 mm.) eureka and nichrome, or antimony and bismuth wires, connected to a heater of flat, thin, platinum ribbon, is supported inside a glass bulb which is joined to the system in which the pressure is needed. A current of the order of 50 mA. is passed through

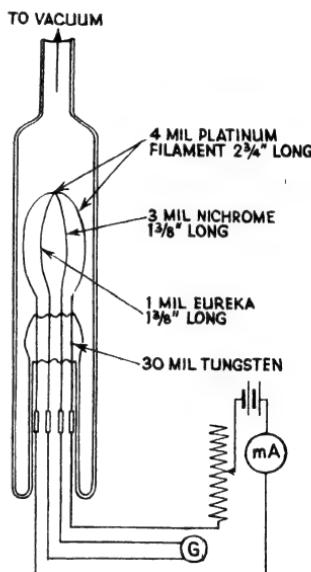


FIG. 78.—The Thermocouple Gauge.

¹ By courtesy of Vacuum Industrial Application Ltd., Wishaw, Scotland.

² J. R. Downing and G. Mellen, *Rev. Sc. Inst.*, 17, 218, 1946.

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the heater, care being necessary not to overheat the junction wires so as to cause them to volatilize. The thermo-e.m.f. developed is recorded by a sensitive galvanometer. Since the temperature attained by the junction depends on the conductivity, and hence on the pressure, so the surrounding gas pressure is recorded in terms of the galvanometer deflection. The useful range of the gauge is between 10^{-1} and 10^{-3} mm. of mercury.

The Knudsen Gauge. An absolute manometer designed by Knudsen needs no calibration against a McLeod. The gauge is delicate and difficult to operate and is more of use in research than in applied vacuum work. It depends on the momentum of impact on a light aluminium vane of heated gas molecules varying with the pressure, the heating being arranged by a filament suspended near the vane in the gas.

Dumond and Pickels¹ have, however, produced a more convenient design of the Knudsen gauge which is satisfactory for routine measurements. Some of the tedious operation involved in the earlier gauges of this type is avoided by making a gauge of less but practically adequate sensitivity, recording pressures down to 10^{-6} mm. Hg. Their construction is shown in Fig. 79.

The Knudsen gauge is considered by these workers to be superior to the Pirani type because it measures the transport of momentum by the molecules of the gas, whereas the Pirani measures the energy of the molecules, which is small compared with the energy of the radiation. Also a limit is set to the sensitivity of the Pirani by the mechanical vibration of the long filaments which have to be used to measure adequately the low pressures.

A light vane is suspended in a chamber joined to the vacuum to be measured. If T_1 is the temperature of the side of the vane exposed to the hot filament, whilst T_0 is the temperature of the other side exposed to the walls of the chamber, then the net displacing force per unit area due to the molecular momentum is given by

$$F = \frac{p}{2} \left[\left(\frac{T_1}{T_0} \right)^{\frac{1}{2}} - 1 \right]$$

where p is the gas pressure. Note that the force F is independent of the molecular weight of the gas.

¹ J. W. M. Dumond and W. M. Pickels, *ibid.*, **6**, 362, 1935.

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In Fig. 79 the aluminium vane is in the form of a rectangular frame 2.5×10^{-3} cm. thick, the inner and outer edges of this frame being folded over to ensure rigidity. This vane is suspended about a vertical axle of aluminium wire, 3×10^{-2} cm. thick, which passes through slits in the frame to which it is spot welded. A 1-cm.

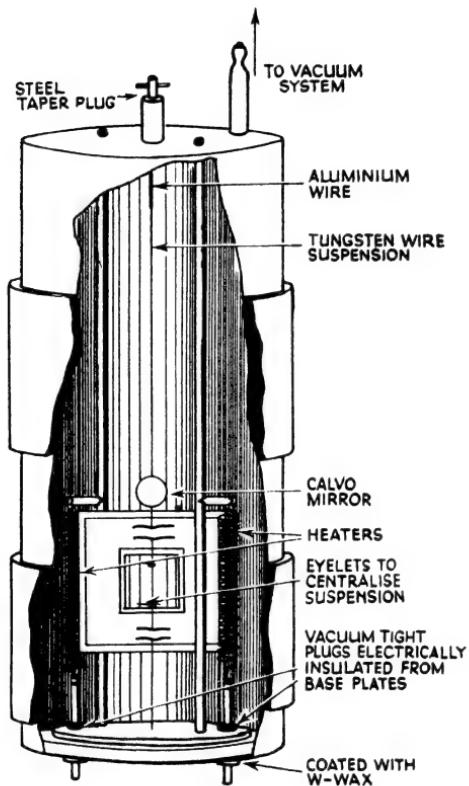


FIG. 79.—A Modified Knudsen Gauge (Dumond and Pickels).

diameter galvanometer mirror attached to this axle permits the angle through which the vane turns to be recorded by the use of a lamp and scale. The suspension wire from the steel taper plug at the head of the instrument is of tungsten. The taper plug acts as a torsion head to enable the angle of the vane to be set; after such an adjustment wax is coated over the join of this plug to the

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top plate to ensure freedom from leakage. The suspension is centralized and prevented from swaying by the eyelets shown. Two nichrome heater wires are arranged on either side of the aluminium frame, one opposite each vertical section. A brass cylindrical envelope encloses the gauge around which water jackets are arranged to ensure a uniform wall temperature. Helmholtz coils, or a permanent magnet suitably disposed about the instrument, are useful to provide electromagnetic damping of the vane.

The Viscosity Gauge.¹ A gauge depending on the variation of the viscosity of a gas with pressure has been designed by Langmuir. The apparatus is simple, consisting of a light quartz fibre oscillating in the gas. The amplitude of oscillation of the fibre varies with the gas viscosity and hence with the surrounding pressure. The gauge is calibrated in terms of the time necessary for the amplitude of the oscillation to decrease by half. The useful range of this gauge is given as 2×10^{-2} to 5×10^{-5} . It is chiefly of use in research on the properties of gases. A pattern designed by the General Electric Co., Ltd., is simple to use. It consists merely of a quartz fibre fixed at the top end inside a glass tube, whilst the lower free end has a small piece of iron attached, which allows the fibre to be set vibrating by simply pulling it to one side of the envelope with a small magnet. The decrement is then observed using a low-powered microscope.

Leak-finding Methods Using Vacuum Gauges. Very useful and sensitive methods of finding small air leaks in vacuum apparatus can be developed if a gauge of the Pirani, thermo-couple, or ionization type is used. Generally a thin jet of hydrogen gas from a cylinder connected to a drawn-down glass tube or to a small "Aerograph" gun, is passed over the walls of the system. Cylinder gas can be used, or a gas rich in hydrogen, such as coal-gas—which will give about one-tenth the sensitivity—or, less satisfactorily, a volatile vapour such as ether or acetone. When the hydrogen stream encounters a small hole or region of porosity in the walls, it will diffuse into the system more rapidly than the denser air, so a sharp increase of pressure recorded is obtained. Thus, using a Pirani gauge, the system is exhausted to an indicated pressure; then, with a sensitivity control in the bridge circuit set at minimum, a resistance is varied to balance the bridge. The sensitivity is then

¹ I. Langmuir, *Jour. Amer. Chem. Soc.*, 35, 107, 1913.

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increased, and a galvanometer of some 100 mm./ μ A. sensitivity switched in place of the normal meter, and the bridge finally balanced. When the hydrogen stream is opposite a leak in the system, the increased pressure is indicated by a sudden movement of the galvanometer spot. This effect will also be enhanced by the greater thermal conductivity of hydrogen compared with air. Leaks in glass or metal systems can be located to within $\frac{1}{8}$ " by this method. For very small leaks, this method is more sensitive if the gauge is in the line between the backing pump and diffusion pump.¹

A leak detector of remarkable sensitivity² has been manufactured by the Radio Corporation of America³: it is the hydrogen-sensitive ionization gauge, or special triode valve R.C.A.1945. Referring to Fig. 80, it is seen that this gauge is essentially a highly evacuated, small, metal envelope, triode valve. The "grid" or ion collector is a cylinder, maintained negative with respect to the cathode, whilst the anode is a palladium plate covering the end of the tube by which the gauge is connected to the system. On heating the cathode by the usual 6.3 V. supply, and maintaining the anode at 185 V., 32 mA., with -22.5 V. on the ion collector, the 6 W. developed at the palladium plate by the electron bombardment will raise it to about 800° C., when it becomes permeable to hydrogen, but to no other gas. When hydrogen, or any gas containing a high percentage of hydrogen, enters a leak in the system from the exploring jet, it will traverse the heated palladium, and cause an increase in the positive ion current of the gauge. When the leak is located, the test gas supply is stopped, and the vacuum pumps restore the gauge to its initial setting by pumping the hydrogen out again through the palladium plate.

To obtain the utmost sensitivity from this method of leak detection, the arrangement shown in Fig. 80b is suggested by R.C.A. A compact instrument incorporating the R.C.A.1945, a regulated power supply, D.C. amplifier, microammeter, valves *A* and *B*, and a vacuum port is supplied. Small electron tubes are checked for leaks by plugging the exhaust stem of each tube into the vacuum port on front of the instrument. An external mechanical rotary pump is then used to exhaust the system to about 10^{-3} mm. Hg,

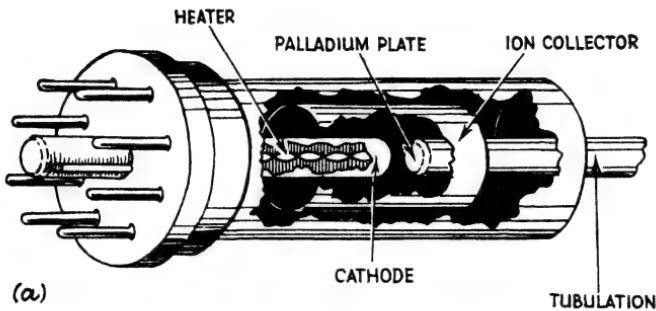
¹ J. B. Kuper, *Rev. Sc. Inst.*, **8**, 131, 1937.

² H. Nelson, *ibid.*, **16**, 273, 1945.

³ Courtesy of R. C. A. Photophone Ltd., Berkeley Square, London, W.1.

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both valves *A* and *B* being open. A hood filled with hydrogen is then lowered over the tube and any leak present is indicated by a sharp upward deflection of the microammeter. On removing the hood, the microammeter returns to its initial setting. To increase the sensitivity of this method for very small leaks, the valve *B* may



(a)

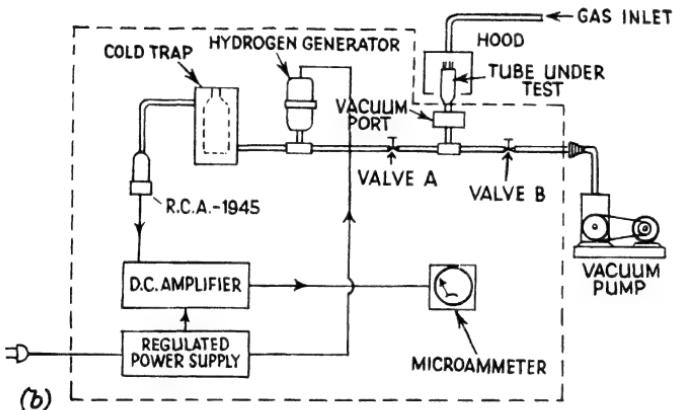


FIG. 80.—(a) The Hydrogen-sensitive Ionization Gauge. (b) System Involving this Gauge as Leak Detector.

be closed after pumping, and then the detecting gas applied; alternatively both valves *A* and *B* are closed, the hydrogen applied, and then valve *A* opened. On the other hand, to use this method for testing leaks in a vacuum system, the system simply replaces the mechanical rotary pump, and the vacuum port on the instrument is plugged. Liquid air or solid CO₂ should be used in the cold trap

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indicated in Fig. 80b. Such a freezing trap is not absolutely essential, but it prevents false indications due to the formation of hydrogen by the action of the hot palladium on the oil vapour from the pump. It is best to let air into the system for two or three minutes prior to using this gauge in order to burn out any oil vapours that may have condensed on the metal walls surrounding the palladium plate.

The hydrogen generator shown in Fig. 80b can be a small auxiliary supply, or in the form of a nichrome filament at 750° C. to 1100° C. to decompose oil vapour, giving hydrogen. Its use increases the sensitivity of this method of leak detection, since the gauge has a maximum sensitivity at a hydrogen pressure of 10^{-2} mm. Hg, which may not be reached by hydrogen diffusing through the leak to be found.

The R.C.A.1945 is supplied with a getter which may be fired by connecting an electric supply at a current of 5.5 A. between pin 8 and the shell of the gauge. This getter should only be used as a last resource : it is intended to reduce the pressure in the gauge if, after prolonged use, it becomes gassy. The pressure in the gauge is normally 10^{-7} mm. Hg.

The outstanding advantage of this ionization gauge method of leak location is its sensitivity : a leak of 10^{-4} litre of hydrogen at a pressure of one micron in one second¹ can be found² ; moreover, the gauge only responds to hydrogen. Thus other leak finding methods employing a Pirani gauge, or normal ionization gauge, may lead to confusion because they respond to gas or vapour released from the walls of the system.

To avoid the necessity of using the special R.C.A.1945 tube, a similar hydrogen sensitive ionization gauge could be constructed based on the arrangement of Fig. 100, page 144, where an electrically heated palladium tube is used in place of the electronically heated palladium plate.

A halogen leak-detector device is the closest rival to the hydrogen-sensitive ionization gauge in providing a reasonably priced sensitive leak indicator. In fact, it is preferred by workers on the Continent

¹ One litre flow of gas at a pressure of one micron per sec. is $l/\mu\text{sec.}$, often referred to as a *lusec*.

² A palladium-barrier leak detector marketed by W. Edwards and Co., Ltd., detects a calibrated leak of 10^{-5} lusecs : a leak rate which would take 25 years to pass 1 c.c. of gas.

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who assert that it gives fewer spurious results. Developed by White and Hickey¹ in America, it has been marketed in England by The British Thomson Houston Co., Ltd.

The operation depends on the fact that the emission of positive ions from a heated anode is markedly increased when the vapours of halogen-bearing compounds impinge on the anode surface. The device therefore consists of a platinum cylinder which is heated in air to about 900° C. by an internal, insulated platinum filament (Fig. 81). Surrounding this cylinder there is a coaxial metal cathode. A P.D. of between 50 and 500 V. is maintained between the anode

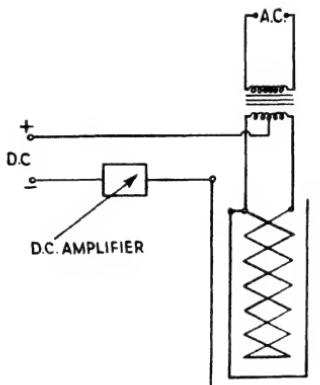


FIG. 81.—The Halogen Leak Detector.

and cathode, a microammeter or resistor with D.C. amplifier being included in series with the supply.

There are two ways of using this assembly, a feature which makes it more versatile than the other methods of leak finding. Firstly, the system in which it is required to find a leak is not evacuated but filled with air containing the vapour of a halogen compound (freon, carbon tetrachloride or trichloroethylene will serve well) so as to be slightly above atmospheric pressure. The air-halogen mixture then issues through any leak, where it may be detected by a narrow tube as a probe which is connected to the electrode assembly. This electrode assembly is often mounted in a metal envelope shaped, for convenience in handling, like a pistol. A rise of the positive ion

¹ W. C. White and J. S. Hickey, *Electronics*, **21**, 100, 1948.

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current as recorded by the microammeter or D.C. amplifier output meter (an alarm bell device may be used) indicates sensitively the presence of halogen. Secondly, and alternatively, the electrode assembly, in its envelope, may be sealed onto the vacuum system in the usual way. Then air containing the vapour of a halogen compound is forced under pressure through a small jet. This jet is then played over the region of suspected leak. If halogen enters the system, the device records its presence and so the presence of a leak. The sensitivity, using a D.C. amplifier, is of the order of 10^{-5} lusecs.

The mass spectrometer used with helium provides the most sensitive and rapid method for the detection of leaks. It can be about 100 times as sensitive as the ionization gauge procedure. The expense of the apparatus prohibits its use except in very large vacuum systems, so it will not be described here.¹ The British Thomson Houston Co., Ltd., supply a mass spectrometer leak detector, using helium gas, which will detect a leak of 10^{-5} lusecs.

¹ For a full treatment, see S. Dushman, *Scientific Foundations of Vacuum Technique*, p. 377, Wiley (Chapman and Hall Ltd.), 1949.

CHAPTER THREE

The Measurement of Pumping Speed

Before describing the practical methods of measuring pump speed, the laws governing the flow of gases through tubes at low pressure must be deduced.

Suppose two parallel surfaces exist at a distance d apart in a gas and the pressure is such that the mean free path is L . Assume that the magnitude of L is much greater than that of d , i.e. so-called *molecular pressures* are concerned. Now, if N is the number of molecules of gas per cubic centimetre and \bar{c} their average velocity then, from Knudsen's cosine law in the kinetic theory of gases, it is obtained that

$$n = \frac{1}{4}N\bar{c} \quad \quad (19)$$

where n is the number of molecules impinging on unit surface in unit time.

Let molecules of gas at these pressures be flowing with a velocity v along a cylindrical tube of diameter D and length l . Consider an element of length δl of this tube; this will have a cylindrical surface area of $\pi D\delta l$. Then the momentum δG transferred per second to the inside surface of this element of the tube by the molecules will be given by

$$\delta G = \pi D\delta l \cdot mv \cdot \frac{1}{4}N\bar{c} \cdot f,^1$$

where f is a numerical fraction depending on the gas and the nature of the walls, and m is the mass of one molecule. Often, for want of experimental evidence, f is taken to be unity. This will be done here.

Let p be the pressure of the gas within the element of the tube and δp the small pressure difference across this element. Equating the force due to this pressure difference to the momentum per second imparted to the cylindrical walls, it follows that

$$\begin{aligned} \frac{\pi D^2}{4} \delta p &= \frac{1}{4} \pi D \cdot \delta l \cdot mv \cdot N\bar{c} \\ \therefore v &= \frac{D\delta p}{mN\bar{c}\delta l} \quad \quad (20) \end{aligned}$$

¹ See M. Knudsen, *Ann. der Phys.*, **28**, 75, 1909.

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whilst the mass μ of gas flowing through the tube element per second will be given by

$$\mu = \frac{\pi D^2}{4} v \rho = \frac{\pi D^3 \delta p}{4mN\bar{c}\delta l} \rho$$

where ρ is the density of the gas at the pressure p .

But $\rho = mN$, therefore

$$\mu = \frac{\pi D^3 \delta p}{4\bar{c}\delta l} \quad . \quad . \quad . \quad . \quad (21)$$

It is sometimes more usual and useful in practice to specify the volume of gas flowing per second rather than the mass. However, this must be done with discretion because the volume of a given mass of gas will depend on the pressure and, in the case of a tube of finite length l , a pressure difference will exist between the inlet and outlet.

Let ρ_1 be the density of the gas at unit pressure—i.e. at 1 dyne/sq. cm. working in C.G.S. units. Then, by Boyle's law

$$\frac{p}{\rho} = \frac{1}{\rho_1} \quad \text{or} \quad p\rho_1 = \rho \quad . \quad . \quad . \quad (22)$$

$$\text{Hence} \quad \mu = \rho V = p\rho_1 V \quad . \quad . \quad . \quad (23)$$

where V is the volume of the mass of gas.

In the case of a tube of finite length l , where p_1 is the pressure at the inlet and p_2 the pressure at the outlet, the mass of gas passing through any cross-section of the tube per second must be constant because there cannot be an accumulation of gas anywhere in the tube. Correspondingly the product pV must be a constant for a given value of $(p_1 - p_2)$.

Putting

$$pV = U$$

U is known as the *throughput* of the tube. Further

$$U = C(p_1 - p_2) \quad . \quad . \quad . \quad . \quad (24)$$

where C is the unit throughput or *conductance* of the tube, i.e. the throughput per unit pressure difference. This will be a constant for a particular tube.

From equations (21), (23) and (24) it follows that, for a tube of length l ,

$$\mu = \frac{\pi D^3 (p_1 - p_2)}{4\bar{c}l} = C(p_1 - p_2)\rho_1$$

\bar{c} being a constant for a particular temperature.

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$$\therefore C = \frac{\pi D^3}{4\bar{c}l\rho_1} = \frac{\pi D^3 p}{4\bar{c}l\rho} \quad . \quad . \quad . \quad (25)$$

on using equation (22).

At a temperature T° absolute,

$$\bar{c} = \sqrt{\frac{3kT}{m}} = \sqrt{\frac{3RT}{M}} \quad . \quad . \quad . \quad (26)$$

where k is Boltzmann's constant, R is the universal gas constant and M the molecular weight of the gas. It is here assumed that \bar{c} is equal to the root mean square velocity \bar{C} . Strictly

$$\bar{c}/\bar{C} = \sqrt{8/3\pi} = 0.92.$$

Further,

$$p/\rho = NkT/Nm = kT/m = RT/M \quad . \quad . \quad . \quad (27)$$

Substituting from equations (26) and (27) in equation (25).

$$\therefore C = \frac{\pi D^3}{4l} \sqrt{\frac{RT}{3M}} = 4,133 \sqrt{\frac{T D^3}{M l}} \quad . \quad . \quad . \quad (28)$$

on substituting $R = 8.314 \times 10^7$ erg deg.⁻¹ mole.⁻¹.

However, a correction is needed to account for the Maxwellian distribution of velocities amongst the molecules. If this is done, the result becomes

$$C = 3,810 \sqrt{\frac{T D^3}{M l}} \quad . \quad . \quad . \quad (29)$$

C being in c.c./sec.

The reciprocal of the conductance C is known as the resistance W of the tube.

In the important case of air, C can be expressed more simply because

$$\begin{aligned} C &= \frac{\pi D^3}{4l} \sqrt{\frac{RT}{3M}} = \frac{r^3}{l} \cdot 2\pi \sqrt{\frac{8.31 \times 10^7 \times 300}{3 \times 28}} \\ &= \frac{r^3}{l} \times 1.08 \times 10^5 \text{ c.c./sec.} \end{aligned}$$

where r is the radius of the tube, the molecular weight of the air, considered as nitrogen, is taken as 28, and T is put as 300° K. = 27° C.

Hence the conductance for a cylindrical tube for air

$$= 10^5 \frac{r^3}{l} \text{ c.c./sec.} = \frac{100r^3}{l} \text{ litres/sec. approx.} \quad . \quad . \quad . \quad (30)$$

whilst the resistance $W = \frac{l}{100r^3} \text{ sec. litre}^{-1} \quad . \quad . \quad . \quad (31)$

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r and l being in centimetres. This convenient and simple formula is adequate for most purposes. In these formulæ, end corrections due to the fact that the gas has to enter and leave the tube are omitted.

The equation (29) demonstrates that the rate of flow gas through a tube at low pressures ($L > D$) is proportional to the cube of the diameter of the tube and inversely proportional to its length. The reader will readily see the necessity for short, wide-bore tubing between the pump and the vessel if a good speed of evacuation is to be obtained.

A simpler case than that of a tube is to consider the flow of a gas through an orifice in a thin wall. The molecular flow case will prevail when the mean free path L is greater than the minimum dimension of the orifice. Suppose the gas on both sides of the orifice is at a uniform temperature T and that the average speed of the molecules is \bar{c} . Let the pressure on one side of the orifice be p_0 whilst that on the other side is p , where $p_0 > p$. The corresponding gas densities are ρ_0 and ρ and the corresponding numbers of molecules per cubic centimetre are N_0 and N .

It is readily seen, using formula (19), that the mass μ of gas which traverses unit area of the orifice per second is given by

$$\mu = \frac{1}{4}N_0 m \bar{c} - \frac{1}{4}Nm \bar{c} = \frac{1}{4}\bar{c}(\rho_0 - \rho)$$

Now
$$p = \frac{1}{3}\rho \bar{C}^2$$

and
$$\bar{c} = \sqrt{\frac{8}{3\pi}} \bar{C} = \sqrt{\frac{8}{3\pi}} \sqrt{\frac{3RT}{M}} = \sqrt{\frac{8RT}{\pi M}}$$

$$\begin{aligned} \therefore \mu &= \frac{\frac{1}{4}\bar{c}(p_0 - p)}{\frac{1}{3}\bar{C}^2} = \frac{\frac{1}{4}\sqrt{\frac{8RT}{\pi M}}(p_0 - p)}{\frac{1}{3}\frac{3RT}{M}} \\ &= \sqrt{\frac{M}{2\pi RT}}(p_0 - p). \end{aligned}$$

The throughput U is, from comparison with equation (24), given by

$$U = C(p_0 - p)$$

C being the conductance of the orifice. Here both U and C are for unit area of the orifice.

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Whilst

$$\mu = U\rho_1$$

where ρ_1 is the gas density at unit pressure.

$$\therefore \mu = \sqrt{\frac{M}{2\pi RT}}(p_0 - p) = C(p_0 - p)\rho_1$$

$$\therefore C = \sqrt{\frac{M}{2\pi RT}} / \rho_1 = \frac{p\sqrt{\frac{M}{2\pi RT}}}{\rho}$$

But $p/\rho = \frac{RT}{M}$ (see equation 27)

$$\therefore C = \frac{RT}{M} \sqrt{\frac{M}{2\pi RT}} = \sqrt{\frac{RT}{2\pi M}}$$

For a circular orifice of diameter D this becomes

$$C' = \frac{\pi D^2}{4} \sqrt{\frac{RT}{2\pi M}} = 2860 \sqrt{\frac{T}{M}} D^2 \text{ c.c./sec.} \quad . \quad (32)$$

on substituting for $R = 8.314 \times 10^7$ erg. deg.⁻¹ mole.⁻¹.

The case of the resistance of a tube of finite length l and diameter D can then be considered as the sum of the resistance denoted by the reciprocal of equation (29), where no end correction is included, plus an end correction equivalent to the orifice of diameter D at the inlet. Hence a useful formula for the resistance W of a tube of length l and diameter D becomes

$$W = \frac{1}{C} + \frac{1}{C'}$$

which, on substituting for C and C' from equations (29) and (32), gives

$$W = \left(\frac{l}{3.810D^3} + \frac{1}{2.86D^2} \right) \sqrt{\frac{M}{T}} \text{ sec. litre}^{-1} \quad . \quad (33)$$

Fig. 82 gives the conductances in litres per second to air at 20° C. for cylindrical tubes of various lengths and diameters. These curves are calculated from equation (33) to give the conductance as the reciprocal of the resistance W .

Gaede's Definition of Pumping Speed.—The speed is measured at a given pressure, and equals the volume of gas pumped from a vessel at that pressure in unit time, where the volume is measured at the pressure.

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Expressed mathematically :

$$S = \frac{-dV}{dt} \quad . \quad . \quad . \quad . \quad (34)$$

where S is the pumping speed and dV is the small change of volume in the short time dt .

Let $R_0 = \frac{1}{S_0}$ be the opposition to the flow of gas at the jet of the pump where S_0 is the pumping speed in litres per second ;

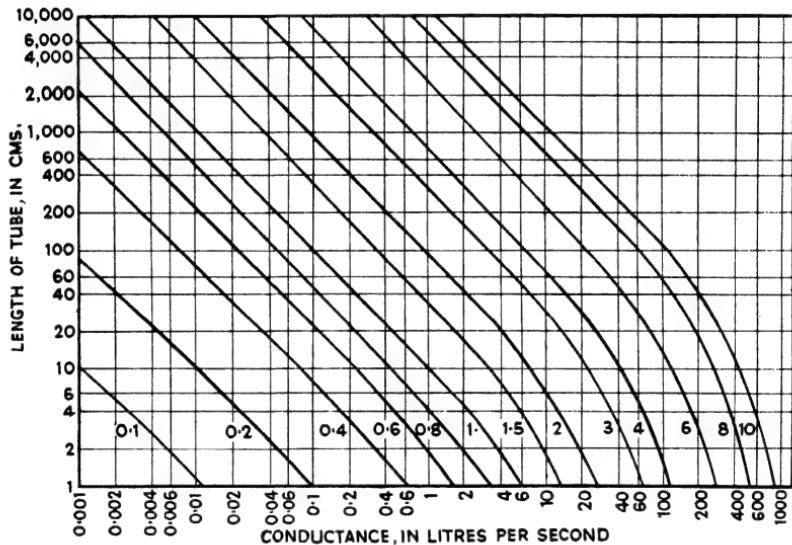


FIG. 82.—Conductance of Cylindrical Tubes for Air at 20° C.
(Parameter is Diameter of Tubes in cm.)

put $R_1 = \frac{1}{S}$ as resistance to flow at the end of a tube of length l and radius r connected to the pump, S being the pumping speed at the end of the tube farthest from the pump. If W expresses the resistance to flow of the connecting tube then it follows that :

$$R_1 = R_0 + W$$

or $\frac{1}{S} = \frac{1}{S_0} + W \quad . \quad . \quad . \quad . \quad (35)$

$$\therefore S = \frac{S_0}{1 + WS_0}$$

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Using the simple equation (31),

$$W = \frac{l}{100r^3} \text{ sec. litre}^{-1}$$

For example, if $S_0 = 20$ litres per second,

$$l = 20 \text{ cm.}, \quad \text{and} \quad r = 1 \text{ cm.}$$

then $W = \frac{l}{100r^3} = \frac{20}{100} = \frac{1}{5}$

Therefore, using equation (35)

$$S = \frac{20}{1 + \frac{1}{5} \cdot 20} = \frac{20}{5} = 4 \text{ litres per second}$$

i.e. the pumping speed is reduced five times. The value of using a fast pump is mitigated unless short wide connecting tubes are used.

Equation (34) gives

$$S = \frac{-dV}{dt}$$

Apply Boyle's law for a perfect gas

$$pV = (p - dp)(V + dV) \quad . \quad . \quad . \quad (36)$$

$$\therefore pdV - Vdp = 0$$

and so

$$\frac{dV}{dt} = \frac{V}{p} \cdot \frac{dp}{dt}$$

making

$$S = \frac{-V}{p} \cdot \frac{dp}{dt} \quad . \quad . \quad . \quad (37)$$

If p_1 and p_2 are the pressures measured at times t_1 and t_2 , then integrating speed over time t_1 to t_2

$$S = \frac{V}{t_2 - t_1} \cdot \log \frac{p_1}{p_2} \quad . \quad . \quad . \quad (38)$$

The pumping speed may be measured applying equation (34), i.e. maintaining the pressure constant and observing the volume change with time; or equation (38) gives the basis of a method in which the pressure change with time is measured keeping the volume constant.

These methods measure the speed of the pump as restricted by any tubing connecting the pump to the measuring gear. Application of equations (33) and (35) can eliminate this factor if the tube dimensions are ascertained.

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The Constant Pressure Method. The vessel *A* (Fig. 83) is being evacuated by the pump under test, and air is admitted to the vessel by the needle valve. An ionization, McLeod, or Pirani gauge, depending on the order of pressure, is connected to the vessel at *T*. The needle valve is adjusted till the pressure as recorded by this gauge is constant. The volume of air pumped out of the tube in time *t* is given by the movement of the mercury pellet¹ along a calibrated capillary tube. This is a convenient arrangement for

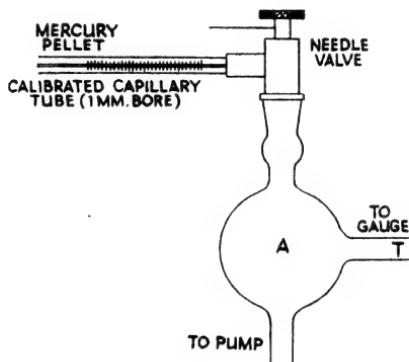


FIG. 83.—The Measurement of Pump Speed at Constant Pressure.

measuring pump speeds at various pressures. The inflow is at atmospheric pressure and the equivalent at the pressure in the vessel is calculated, using Boyle's law.

The equation

$$S = -\frac{dV}{dt} \quad . \quad . \quad . \quad . \quad (34)$$

applies, giving the pumping speed, which can be obtained directly or by plotting a graph of volume against time and obtaining the tangent of the slope. This graph should, of course, be a straight line, since the pressure is constant.

The constant pressure method should always be used for high-speed pumps in which the pumping speed varies greatly with the

¹ A. Leemans suggests the use of a close-fitting steel ball lubricated with silicone oil in place of the mercury pellet. Such a ball can be moved to any desired position along the capillary tube by the use of a magnet. (*Private communication.*)

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pressure. A family of lines of V plotted against t with P as parameter, from which a speed against pressure curve is derived, are necessary to illustrate the action of the pump over a wide range of varying pressure conditions.

An alternative constant pressure method is to apply (33) equation for the flow of gas through a tube (Fig. 84).

Two bulbs of the same capacity terminate the flow tube of which the dimensions are known. One bulb is at pressure p_1 , the other at p_2 . p_1 is the fore-vacuum pressure, and p_2 the final high-vacuum pressure. Both bulbs are furnished with a sensitive gauge. A McLeod gauge can be used as the pressures are steady and the effect of the connecting tube to the gauge is unimportant.

The resistance and hence the conductance of the tube can be

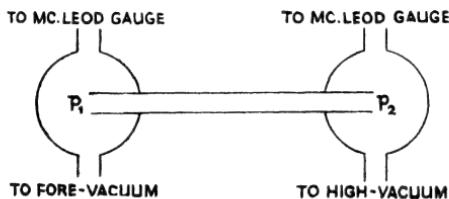


FIG. 84.—Alternative Constant Pressure Method.

calculated from equation (33) where the temperature T is known and M is 28. Alternatively, the conductance can be determined from Fig. 82. Pure dry air must be used.

The throughput of the tube U is given by equation (24) as

$$U = C(p_1 - p_2)$$

Now $U = p_2 V$, where V is the volume of air flow per second at the pressure p_2 . This volume flow per second will be the speed S of the pump required, which is specified as at the high vacuum pressure p_2 . Hence

$$\begin{aligned} U &= p_2 V = p_2 S = C(p_1 - p_2) \\ \therefore S &= \frac{C}{p_2}(p_1 - p_2) \quad . \quad . \quad . \end{aligned} \quad (39)$$

H. C. Howard¹ describes a modification of the constant pressure method which is more suitable for pumps of great speed such as

¹ H. C. Howard, *Rev. Sc. Inst.*, **6**, 327, 1935.

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large-throat mercury diffusion pumps. His apparatus is shown in Fig. 85. In use the stop-cock *C* is closed at first and the level of the mercury in the burette lowered till there is free communication to the atmosphere through stop-cock *B*. The upper stop-cock *C* has a nicked bore, so that its aperture size can next be carefully adjusted until the pressure at the mouth of the pump, as recorded by the McLeod or other gauge, reaches the required value. After about twenty minutes the pressure will become constant, and the level of the mercury in the burette is raised until communication with the atmosphere is cut off and stop-cock *B* is closed. The

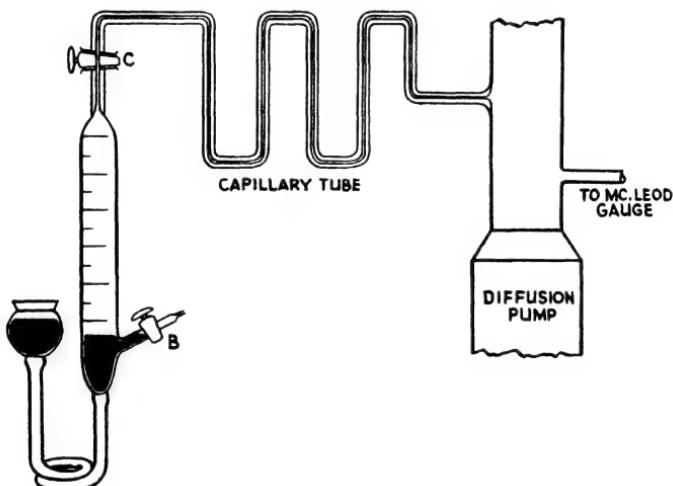


FIG. 85.—Measurement of Pump Speed (H. C. Howard).

volume of the air in the burette, the time and the atmospheric pressure are recorded. The pump then draws out the air from the burette, whilst the pressure is kept approximately atmospheric by adjusting the height of the levelling bulb. After a suitable time, say two minutes, the burette reading is recorded. In this way the volume of air pumped per second at any required pressure at the pump nozzle can be obtained.

Downing and also Dushman¹ describe a method of measuring pump speeds at constant pressure using an oil pipette in the form

¹ See S. Dushman, *Scientific Foundations of Vacuum Technique*, Wiley (Chapman and Hall Ltd.), 1949.

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of a U-tube (Fig. 86). The disadvantage of oil is that it is liable to release dissolved gas. The low vapour pressure silicone or Apiezon oils may be used, though butyl phthalate is often preferred because it does not "wet" glass to the same extent. The advantage of using oil over mercury is its comparatively low density. The tap T of Fig. 86 is first open so that the pressure in the U-tube is atmospheric and the oil levels are equal. The rate of leak of air

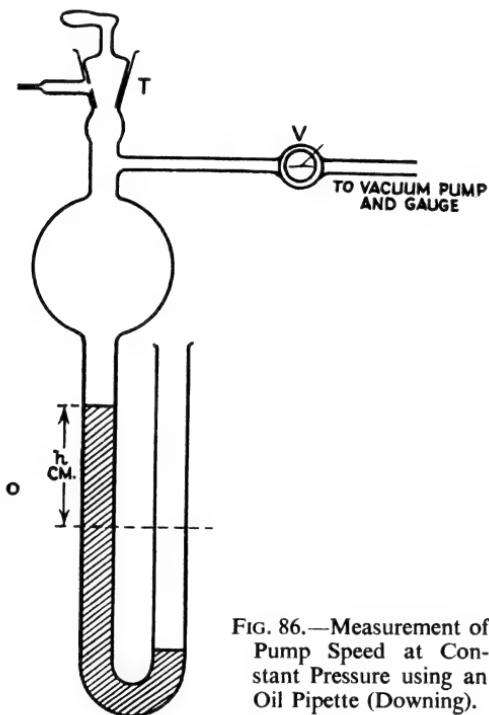


FIG. 86.—Measurement of Pump Speed at Constant Pressure using an Oil Pipette (Downing).

into the pump is adjusted by the needle valve V until the required constant pressure, as recorded by an attached gauge, prevails at the vessel connected to the pump. When an equilibrium pressure is established, the tap T is closed. The level of the oil in the left-hand limb of the U-tube will then rise whilst that in the right-hand limb falls. The time of rise of the oil through a distance h cm. is measured. The needle valve between the U-tube and the pump is then closed so that the rise of oil ceases. Opening the

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tap T to the atmosphere again re-establishes equal oil levels, and the measurement can then be repeated.

If α is the area of cross-section of the uniform bore U-tube, then ha is the volume of air removed from this tube in the measured time t . The pressure in this tube is initially atmospheric and finally atmospheric pressure minus $2h$ in centimetres of oil. Thus if the atmospheric pressure is 76 cm. Hg, whilst the density in grams per cubic centimetre of the oil is ρ , then the mean pressure in the U-tube is $(76 - \frac{hp}{13.6})$ cm. Hg, where ρ is approximately unity.

If h is not greater than 13.6 cm., the error introduced by assuming the pressure in the U-tube to be atmospheric throughout the experiment is not greater than 1 in 76. A correction can be applied for this small departure from atmospheric pressure (Dushman, *ibid.*).

Neglecting this correction, then if the atmospheric pressure is B mm. Hg, whereas the pressure recorded by the gauge at the vessel connected to the pump is p mm. Hg, then the speed S of the pump is given by

$$S = \frac{h\alpha}{1000t} \frac{B}{p}$$

at the pressure p , where h is in centimetres, α in square centimetres and S is given in litres per second if t is in seconds.

The Constant Volume Method is not such a satisfactory procedure as the constant pressure method because all pump speeds vary with pressure. Equation (38) is applied, a vessel of known volume being pumped for the time t_1 to t_2 and the pressure change p_1 to p_2 recorded by the most convenient gauge depending on the pressure attained, except that the McLeod must not be used because of the time-lag introduced by the tube connection to the gauge, which has appreciable resistance to gas flow.

In all the above methods the restricting effect of the connecting tube must be considered applying equations (33) and (35).

P. J. Mills¹ used the apparatus of Fig. 87 to investigate the time-pressure characteristics of various pumps. His results are of interest considered in connexion with the subject matter of Chapter One. Pumping a volume of 4000 c.c. and recording pressures with the McLeod gauge, a "Hyvac" pump reached 8×10^{-5} mm. in 80 minutes, a "Megavac" reached 1.1×10^{-4} mm. in 47 minutes,

¹ P. J. Mills, *Rev. Sc. Inst.*, 3, 309, 1932.

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and a "Hypervac" attained 5.4×10^{-5} mm. in 40 minutes. He verified that a single-stage diffusion pump has a speed and a final pressure which are sensitive to fore-pressure values, whereas a three-stage pump showed little variation with comparatively large fore-pressure changes. The molecular pump, as would be expected on considering equation (3), page 13, behaved much like a multiplier in its action to the fore-pump. Using an ionization gauge, diffusion pumps employing Apiezon oils failed to register a pressure lower than 3.5×10^{-4} mm. Hg unless a carbon dioxide "snow" trap was used. A moderate freezing trap such as ice with brine was of no benefit in this connexion. The pumping speed of activated

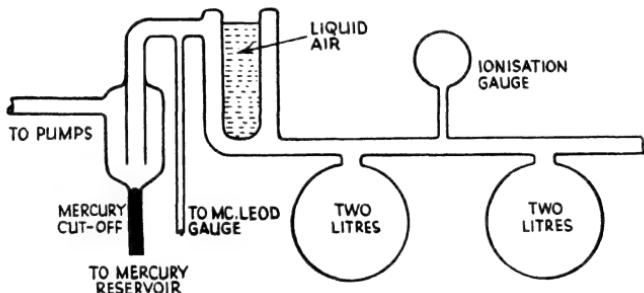


FIG. 87.—Determination of Time-Pressure Characteristic of a Pump.

coconut-shell charcoal was also investigated. A single-stage, air-cooled, horizontal diffusion pump, using Apiezon B oil was employed, backed with a "Megavac". Eighty grams of charcoal were introduced into the system, the pumps started, and the charcoal baked at 480°C . for four hours. The diffusion pump was then switched off, and nitrogen introduced until the pressure was atmospheric, and then the charcoal-filled side-tube was surrounded with liquid air. A pressure of 5×10^{-2} mm. Hg was recorded. On standing overnight without liquid air the charcoal re-evolved the nitrogen till the pressure rose to 20 cm. Hg. As a second experiment, after the pumps were started, the large volumes and charcoal trap were baked for an hour at 400°C . and 430°C . respectively, the pressure meanwhile being about 10^{-2} mm. The system was then cooled to room temperature, when, in less than an hour, a pressure of 3×10^{-6} mm. was attained.

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Precautions in Measuring Speeds. Dayton¹ makes some observations of great importance in connexion with the determination of speed, especially significant in measuring the performance of large diffusion pumps. He states that errors of as much as 50% are easily obtained if the gauge position and air inlet location are not correctly chosen. Thus in the normal use of a pump, connected to a chamber of considerable size, air enters the throat at all angles, and some of the molecules strike the side walls or top cap in the jet assembly, and are then scattered back out of the pump. On the other hand, when air issues from the small hole used as a leak in measuring speed, then molecules may be beamed directly into the vapour jet, or otherwise aided. Again, if the gauge is directly opposite the air inlet, the pressure reading will be higher than normal, and the measured pump speed less than the true value, whilst if the incoming air is directed away from the gauge, an opposite effect occurs. Hence Dayton insists that, for true recording, a test dome of diameter at least equal to that of the pump casing and of height as great as the diameter should be used, with the air inlet introduced at a point high on the side of the dome. The gauge is then central in this dome. According to Dayton, any pump which gives a speed indicating a H_o coefficient of more than 50% is open to criticism of the speed-measuring practice. Thus the closing of the unbaffled pump with a flat plate into which are inserted the gauge and the air inlet is strongly deprecated as practice giving a misleadingly high figure, especially in the case of a diffusion pump with inlet diameter greater than 3".

The selection of a gauge for use in measuring the pressure during speed measurements should be done with the following points borne in mind :

(1) A McLeod gauge is satisfactory in a constant pressure method if sufficient preliminary test is made of leak rate against pump speed to ensure that the slow reading McLeod is recording a constant pressure during the time of the experiment.

(2) An ionization gauge may give too high a reading of the pressure if directly exposed to oil from a diffusion pump because the oil may disintegrate under the action of the hot gauge filament.

¹ B. B. Dayton, *Ind. and Eng. Chem.*, **40**, 795, 1948. See also S. Dushman, *Scientific Foundations of Vacuum Technique*, Wiley (Chapman and Hall Ltd.), 1949.

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(3) Mercury vapour from the burette or levelling device will give a reading of its vapour pressure on all gauges except the McLeod, unless suitable traps are present. Oil, such as Apiezon B, can be used in place of mercury, but is liable to give much trouble due to occluded air. Perhaps the best way of using oil at the air inlet device is to replace the mercury burette by a calibrated glass tube dipping vertically into a tall jar of oil, and move either the tube or jar to ensure that the final levels of oil inside and outside the tube are the same.

(4) It is only gauges such as the Knudsen or the McLeod for perfect gases which give readings of low pressures which are irrespective of the nature of the gas molecule. This should be considered if speed measurements for a gas other than air are required, e.g. speed for hydrogen, or for the residual gases and vapours given off by the walls of a vessel.

CHAPTER FOUR

De-gassing and "Gettinger"

The removal of air and gases from vessels is accomplished by the methods described in the first chapter. The free gases, i.e. gases not adsorbed or absorbed by the materials of the vessel, are removed until the prevailing pressure is as low as 10^{-6} mm. Hg corresponding to a mean free path of 5000 cm. If the vessel is being continually pumped this is a low enough pressure for most vacuum-tube operations. In many types of apparatus, e.g. electric lamps, valves, cathode-ray tubes and so on, the vessel has to be sealed off from the pump, and during a life of several hundred hours (a thousand hours is the usual minimum life of a radio valve) the pressure must not rise above 10^{-4} mm., corresponding to a mean free path of 50 cm. If the pressure rises to 10^{-3} mm., then the mean free path is 5 cm., comparable with the dimensions of a small tube, and ionization by collision takes place in a tube in which free electrons with above ionization energies are present. To exhaust a vessel which has to maintain so low a pressure after sealing off from the pump requires that any gases adsorbed (as surface layers) on the glass walls and metal structures in the vessel must be evolved by heating, so as to be able to pass freely to the pump during exhaustion, whereas absorbed gases (in the body of the substance) need a still higher temperature to release them.

In general there are four methods of releasing these gases. Firstly, by baking the whole vessel in an electric or gas oven up to a temperature just below the lowest softening point of any material in the tube (usually the glass envelope). Secondly, material inside the vessel, if the envelope is an electrical insulator, can be raised to red or white heat by a process of radio-frequency eddy-current heating providing the material is a conductor of electricity, i.e. the metals and carbon ; the temperature must not be above the point where the vapour pressure of the metal is greater than 10^{-7} mm. Hg, otherwise volatilization of the metal will begin. In the third case a "gettering" process is used whereby a chemically active metal

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like barium or magnesium is volatilized by electrically heating it in the tube after the pumps have attained the lowest possible pressure, and just before sealing off the tube. These "getters", during volatilization, combine with the residual gases in the vessel so that they are fixed as chemical compounds deposited on the walls of the tube. Moreover, some "getters", particularly barium, will adsorb small quantities of gas released after the tube is sealed off the pump. The fourth method is to employ what is known as discharge cleaning, or the removal of surface layers of vapour or grease on materials *in vacuo* by bombardment with positive ions.

Each of these four methods is discussed in detail below. Their technical application to particular types of tubes is dealt with more fully in the following chapter.

Baking the Vacuum Tube in a Low-temperature Oven. This method is, of course, applicable only when the vessel material is not affected by temperature rise; the commonest examples are glass and silica tubes. Electric heating and gas heating are both used. The oven is usually made of asbestos or one of the commercial asbestos-cement mixtures, and reinforced by a skeletal structure of steel straps. Using electric heating, a coil of "brightray" wire is wound inside the oven, the wire being uniformly spaced over the whole inside area of the oven so as to maintain as uniform a temperature as possible. A current of 20 to 25 amps. at 220 V. gives about 5 kW. of electric power suitable for raising an oven of dimensions about $2' \times 1' \times 1'$ to about 600° to 700° C. Such an oven is suitable for baking experimental electron tubes such as cathode-ray tubes and X-ray tubes. The oven is best supported from a pulley using a counterpoise weight, its up-and-down motion over the tube being guided by runners. Fig. 88 illustrates such an arrangement; PP_1 is the pulley system, GG_1 being runners to prevent sideways motion of the oven and the danger of breaking the glass tube. At T a thermocouple is inserted through a hole in the oven for temperature reading. It is often useful to provide a mica window in the oven walls to observe the tube during the baking process.

Gas-heating is preferred by some vacuum workers. The ring-shaped ovens on multiple valve-pumps are often gas-heated (see Chapter Five). Numerous gas-jets arranged around the base of the inside wall of the oven provide a readily controlled means of

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obtaining a uniformly dissipated heat over a temperature range from room temperature up to 1000° C.

The baking must not be commenced until the pressure in the vessel is below 10^{-3} mm. Hg. Then the oven is lowered carefully over the tube and its temperature raised. Care must be taken to ensure that this temperature rise is not too rapid if soft glass vessels with much strain in them are being baked. For baking up to 250° C. a mercury thermometer is the best indicator of temperature;

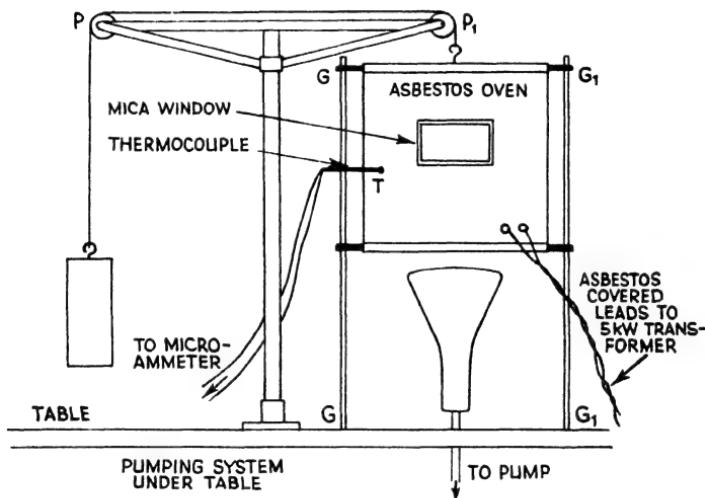


FIG. 88.—Oven for Baking a Vacuum Tube.

above this temperature a thermocouple is used. The baking time and temperature are determined by the nature of the glass and the size of the vessel. Lead glass can be heated to 400° C., soda glass to 450° to 500° C., Pyrex glass to 600° to 650° C. A normal-size receiver valve requires 10 to 15 minutes baking. A vessel 8" in diameter needs a bake of about one hour. A 12"-face cathode-ray tube should be baked for about 2 hours to ensure adequate removal of gas from the glass.

At 150° C. the greater part of adsorbed water-vapour and gases is given off glass surfaces in vacuum. The unimolecular gas film which invariably adheres to glass surfaces is released at 300° C.

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The rates at which gases are evolved from glasses at various temperatures are indicated by the following table of figures :¹

Glass	Cubic mm. of total gas given off by glass surface area of 350 sq. cm. at temperatures—						
	100° C.	150° C.	200° C.	300° C.	400° C.	500° C.	600° C.
Soda glass	0	36	22	8	6	11	50
Lead glass	6	10	10	7	8	12	30
Borosilicate glass	4	20	20	10	16	12	30

Gases evolved are chiefly water vapour and carbon dioxide.

The structure inside the glass or silica envelope may decide the maximum safe baking temperature. For example, a photo-cell containing free caesium cannot be heated beyond 180° C. The baking should be continued until the pressure recorded by the gauge is the lowest obtainable by the pump used (usually between 10^{-5} and 10^{-6} mm. Hg). In this connexion it is important to realize that the rate at which gas is evolved from a vessel is a large factor in determining the necessary pumping speed of the system, otherwise such large pressure increases may occur during baking, that surfaces inside the electron tube, e.g. photo-cathodes and thermionic cathodes, may be ruined.

Out-gassing of Metals. Before metal or carbon electrodes are inserted in an electron tube they must be out-gassed as thoroughly as possible. This is best done in a vacuum stove ; Fig. 89 illustrates the apparatus.

A silica tube of suitable dimensions, wall thickness about 8 mm., is pumped by a backing and diffusion pump combination (a backing pump only is satisfactory for many metals, e.g. a Megavac or Edwards Speedivac pump). One end of the tube is sealed off, the other end is closed by a steel plate. This steel plate is water-cooled and fits flat on the metal flange cemented to the end of the tube. A rubber gasket between the flange and plate will ensure freedom from air-leaks. The heating is by 16 s.w.g. nichrome wire supplied with 5 to 10 kW. of electrical power (for a tube of dimensions indicated in Fig. 89). Alternatively, a series of gas burners round the silica tube inside a well-lagged container can be used. The

¹ Sherwood, *Jour. Amer. Chem. Soc.*, **40**, 1645, 1918.

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lagging is asbestos, fire-brick, or Kieselguhr. The metal electrodes to be out-gassed are put in a nickel or molybdenum boat and inserted in the stove. The pressure must be less than 10^{-8} mm. Hg. A "black" discharge in a discharge tube some 3" long with an

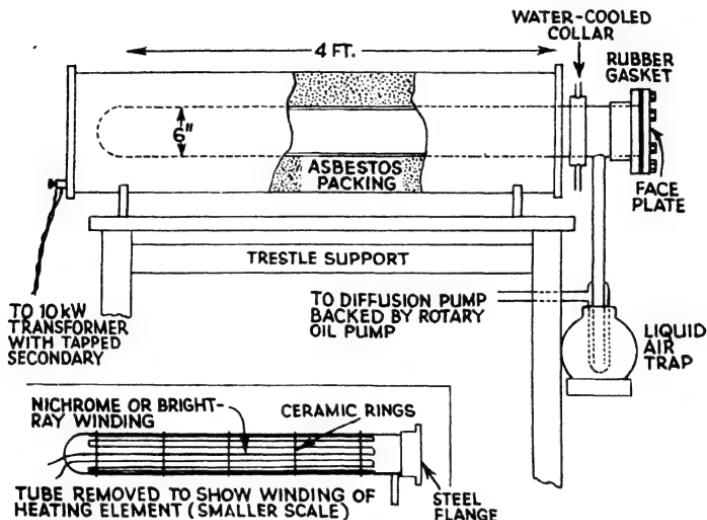


FIG. 89.—A Vacuum Stove.

applied potential difference of 15 kV. is a sufficiently good indication of pressure needed.

The out-gassing temperatures for the common metals are given in the following table :

Metal	Out-gassing temp. (max.) in ° C.
Tungsten	1800
Molybdenum	1760 *
Tantalum	1400
Platinum	1000
Copper and Cu alloys	500
Nickel and Ni alloys	750-950
Iron	1000
Graphite	1500-1800

* Though molybdenum is best heated to 1760° C. to out-gas it thoroughly, yet it becomes brittle if heated at temperatures in excess of 950° C.

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Some metals, e.g. molybdenum, platinum, nickel and iron, may be cleaned by heating in a hydrogen atmosphere.

Using a heater coil of tungsten wire coated with zirconium oxide insulation a small stove capable of reaching 2000° C. can be constructed.¹

Eddy-Current Heating. If a coil carrying a large radio-frequency current is placed near a conductor in vacuum the eddy currents induced in the conductor will raise its temperature. The same effect occurs at atmospheric pressure, a fact which the operator should realize if small metal objects are in the neighbourhood, but obviously the temperature rise is much less.

This fact is of great use in raising to high temperatures the metallic or carbon structures used in electron tubes.

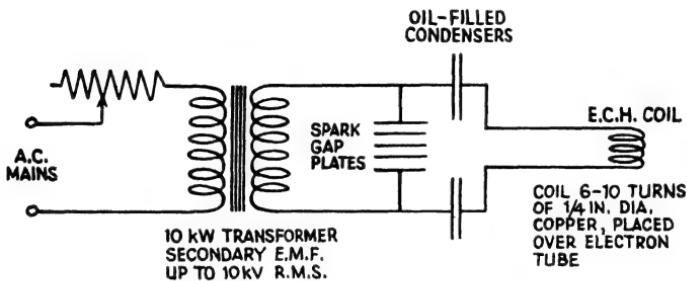


FIG. 90.—Basic Circuit of a Spark Eddy-current Heater.

Thus the nickel anode of a wireless valve, or the electron-gun in a cathode-ray tube may be raised to 800°-900° C., and gases which were not evolved by the oven-baking will be rapidly released. Again, the pumping speed must be sufficient, otherwise the gas evolved may ionize to give a discharge under the excitation of the radio-frequency wave.

Typical circuits for producing such oscillatory currents are shown in Figs. 90 and 91.

Fig. 90 is the circuit of a spark oscillator absorbing up to 10 kW. of power and giving in the output coil up to 50 amps. of current at a frequency of some 100 kilocycles. The spark eddy-current heater is very robust and reliable. Care must be taken to prevent

¹ See *Werkstoffkunde der Hochvakuumtechnik*, by Espe and Knoll (Julius Springer).

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interference with broadcast reception as the output contains strong harmonics of the fundamental frequency.

Fig. 91 is a valve oscillator circuit. Frequencies up to 1000 kilocycles can be used. It has the advantage that it is more readily controlled. The circuit represents a simple apparatus: bigger output would require four or six valves operated in parallel with more rectifying valves. A push-pull arrangement of two valves would give greater efficiency.

The leads to the output coil must be of stranded copper cable or strip of ample cross-section, otherwise skin resistance losses will

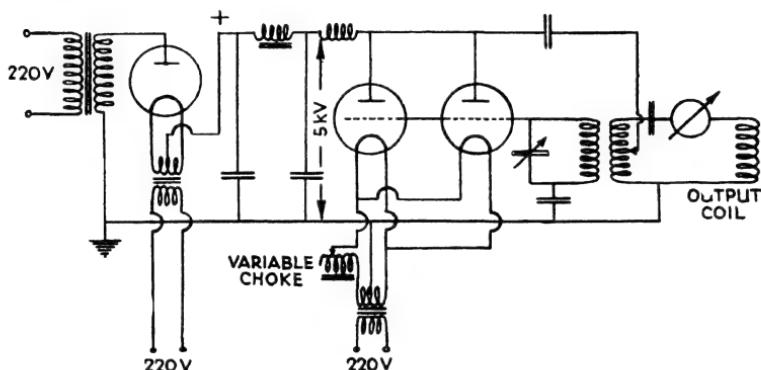


FIG. 91.—Basic Circuit of a Valve Eddy-current Heater.

be high. They must be water-cooled. The coil carrying the radio-frequency current, which is placed over the electron tube to heat the electrode structure inside, is of copper rod, some $\frac{1}{4}$ " in diameter, or copper tubing through which water flows. The coil must be supported by a long insulated handle for safe operation. See Fig. 92.

The higher the frequency of the current in the output coil the greater is the efficiency of coupling or transfer of electric energy in the coil to heat energy in electrode. A practical limit is set, however, by the fact that the output coil will have a voltage $2\pi f L I$ across it where f is the frequency, I the current and L the inductance of the coil in henries. If f is too large, then this voltage may be so great as to cause sparks to jump across the turns of the coil. A million cycles is the extreme upper limit usable, whereas

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500–600 kilocycles is a normally used practical figure. This factor will also influence the spacing of the coil turns. A usual arrangement is for coil to have about $\frac{1}{4}$ " between adjacent turns.

The time of eddy-current heating depends very much on the particular electron tube: no definite rule can be stated even for electrodes of the same size. The anode of a valve will require a heating time depending on the cathode emitter, and its tendency to out-gas. A cathode-ray tube gun will need about a ten-minute heating, but again this will vary with the size of the gun, the "getter", the size of the tube and the disposition of the electrodes in it. Time schedules for such heating have to be worked out in conjunction with a sensitive ionization gauge so that it is known that

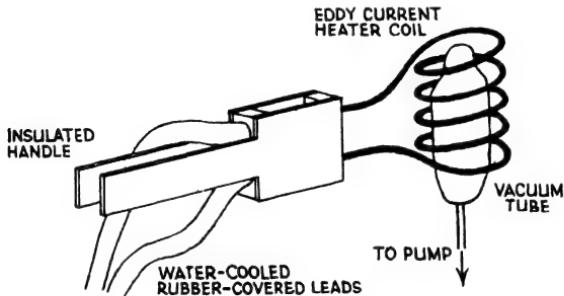


FIG. 92.

for a certain electrode tube, then the out-gassing will be below a minimum rate after a determined time. The temperature is as high as possible, yet avoiding any volatilization of metal atoms from the heated electrode. If too furious an out-gassing occurs giving rise to an intense discharge glow excited by the electromagnetic radiation, then the heating must be temporarily stopped.

Equations relating the radio-frequency energy N_T in watts necessary to raise metals to a specified temperature T° abs. have been discussed by Reche.¹ He gives the equation

$$N_T = 2 \cdot 8 \times 10^{-4} A \sqrt{f \cdot \mu \cdot \rho_t} (nI)^2 \text{ watts} . . . (40)$$

for a cylindrical electrode heated *in vacuo*. A is a constant depending on h the height of the cylinder, d its diameter and D the diameter of the eddy-current heater coil (the thickness of metal of the

¹ Reche, *Wiss. Veroff. Siemens-Konz.*, **12**, 1, 1933.

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cylinder is negligible if below about 0.3 mm.) ; f is the frequency of the current in the coil ; μ is the magnetic permeability of the electrode (equals one in most cases as temperature above 700° C.) ; ρ_t is specific resistance of the metal heated in ohm-cm. ; n is the number of turns in coil and I the R.M.S. value of current flowing in amperes.

At a temperature T° absolute a surface of area a sq. cm. radiates energy according to equation

$$N_T = 5.75 \times 10^{-12} \cdot T^4 \cdot a \text{ watts} \quad . \quad . \quad . \quad (41)$$

following the theory of black-body radiation which applies approximately to most metals above 800° C. Equation (40) can be written in the form

$$nI = \sqrt{\frac{N_T}{2.8 \cdot 10^{-4} \cdot A(f \cdot \rho_t)^4}} \quad . \quad . \quad . \quad (42)$$

The constant A is given by the following table. Intermediate values may be found approximately by interpolation (an approximate value of the current is all that is required).

$\frac{D}{d}$	$\frac{d}{h}$	A
1.2	1.0	1.6
	0.5	0.9
	0.2	0.4
1.8	1.0	0.9
	0.5	0.7
	0.2	0.3

where D = diameter of eddy-current heater coil

d = diameter of cylindrical electrode

h = height of cylindrical electrode.

As an example, consider a cylinder of nickel of height 5 cm., diameter 2.5 cm., and therefore of surface area 39 sq. cm., to be heated to 800° C. (1073° absolute).

Applying equation (41), then

$$N_T = 5.75 \cdot 10^{-12} \cdot (1073)^4 \cdot 39 = 300 \text{ watts approx.}$$

At 800° C., $\rho_t = 0.45 \cdot 10^{-4}$ ohm-cm., and suppose frequency of

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the current equals 10^6 cycles per second, in a coil of 3.0 cm. diameter then

$$nI = \sqrt{\frac{300}{2.8 \cdot 10^{-4} \cdot 0.9 (10^6 \cdot 0.45 \cdot 10^{-4})}}$$

from equation (42) since $A = 0.9$ when $\frac{d}{h} = 0.5$ and $\frac{D}{d} = 1.2$ from above table

$$= \sqrt{\frac{300}{1.69 \cdot 10^{-3}}} = 425$$

i.e. a coil of 10 turns would require a current of 42.5 amps. R.M.S. at 10^6 cycles per sec.

"Gettering"—a process employing the chemically active alkali and alkali-earth metals whereby they are heated till they vaporize in the vacuum and combine chemically with any residual gas in the vessel. A second process is also employed to a minor extent in which the absorption for gases of certain hot metals is used.

Barium. The best and most used getter is barium. It is supplied commercially in the form of the free metal coated with nickel or copper to form a pellet some 3 mm. long by 2 mm. diameter. A narrow tube of copper is filled with barium, cut into pellets and the open ends of the pellet tubes sealed over.

To heat the getter in vacuum this pellet is mounted on a small disc or cup of out-gassed nickel and heated by eddy-currents at about 700°C .; the high vapour pressure of the barium will cause it to break through the copper coating. Suitable arrangements are depicted in Fig. 93.

This small assembly is welded to a suitable part of the electrode system of the vacuum tube where raising its temperature by eddy-current heating will not simultaneously heat another portion of the electrode structure undesirably, and also so that, with suitable shielding arrangements, the getter metal is not splashed on parts of the tube across which there should be high insulation. Fig. 94 shows a common receiver valve assembly in which the getter is well clear of the electrode system by its insertion near the valve pinch. This pinch is sometimes sand-blasted to avoid the deposited metal getter forming a continuous conducting film across it. The eddy-current heater coil is sometimes a flat form spiral. Care must be taken to avoid heating the deposited getter film on the glass,

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otherwise the bulb may crack or "suck" in. At an early stage in the activation of the tube, it is desirable to pre-heat the getter and deposit a small portion of it, then it is ensured that the final

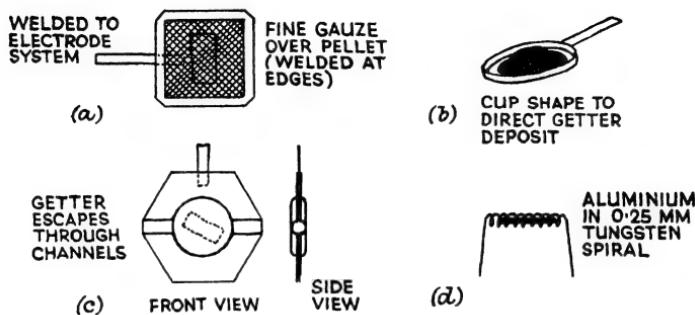


FIG. 93.—Getter Supports.

gettering process just before the tube is sealed from the pumps will not simultaneously evolve gas during the "cracking" of the getter, whilst the getter vapour is endeavouring to trap the normal residual gases. If a glow discharge forms during gettering or pre-gettering

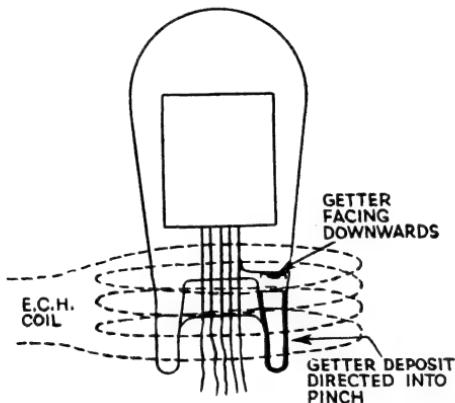


FIG. 94.—Getter in a Valve.

due to evolved gases being excited by the high-frequency radiation from the coil, it is advisable to stop heating the getter and allow the diffusion pump to draw off the excess gas before continuing the

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gettering process. This gas discharge commonly occurs when automatically gettering mass-produced receiver valves. If a rapid getter, of high " pumping speed ", is used, its presence is not necessarily harmful.

The pellets should be kept in small evacuated bulbs until used.

A temperature of 700°–800° C. ensures a rapid evaporation of barium. All but the rare inert gases—argon, neon, krypton, xenon, helium—will be absorbed or adsorbed. The deposit formed on the walls is of a brownish-silver colour. It has the outstanding advantage that it will continue to adsorb gases after the evaporation is complete, and even after the vacuum tube has been sealed off from the pump for several days.

In the mass production of radio receiver valves a backing pump only is used ; the manufacturer relies on the " getter " to produce a final vacuum of less than 10^{-5} mm. Hg.

Magnesium is a useful getter material. A strip of magnesium ribbon some 4 mm. by 2 mm. mounted on a disc of nickel will satisfactorily absorb, when heated to 800° C., oxygen, nitrogen and carbon dioxide. The deposit is bright silver in appearance. It does not adsorb any appreciable amount of gas after deposition.

Calcium. Though not so effective a getter as magnesium or barium, calcium has the advantage that the deposited calcium salt is non-conductive. If freedom from electrical leaks is essential in the tube, this is a considerable asset ; an example of its use is in the special valves used for microphone amplifiers in which a very low " noise " level due to random electron paths is necessary. Calcium chips, free of oxidation, are mounted on a metal disc and welded in position under a fine wire gauze, the gettering being done by eddy-current heating.

Aluminium. A piece of 1-mm. pure (99.9%) aluminium wire heated by electric current passing through a surrounding tungsten spiral will getter oxygen, carbon dioxide and nitrogen. This application is sometimes used in reducing the pressure in large vacuum vessels, e.g. when aluminizing mirrors. Aluminium is not used in electron tube work as a getter.

Zirconium, tantalum and tungsten wires heated by electric current in a vacuum exhibit the useful property of absorbing or evolving gases depending on their temperature. Thus at 800° C. tantalum will absorb 700 times its own volume of hydrogen, whilst at 1100° C.

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it will re-evolve it. A controllable supply of hydrogen can be obtained in this manner. Oxygen and nitrogen are likewise absorbed. Tungsten will absorb oxygen strongly at 1000° C. if the prevailing pressure is not above 10^{-2} mm. Hg. Nitrogen and carbon monoxide are absorbed, giving tungsten nitride and tungsten-carbon-oxygen compounds, at 2300° C.

Zirconium¹ operated at 1400° C. getters oxygen, nitrogen, carbon monoxide and carbon dioxide, whereas at 300° to 400° C. it absorbs hydrogen. Hence, for complete gettering, two wires, one at 1400 C. and the other at 400° C., are necessary. May be applied as a fine powder on anode of a transmitting valve and heated to required temperature by electron bombardment from the emitting filament.

Drying Agents. The removal of water-vapour from a vacuum system is of great importance ; the mechanical backing pump will not cope with it, unless a gas-ballast type is used. A glass trough containing a drying agent placed in a suitable part of a pump system will usually improve considerably the performance. The usual position is in the vacuum line between the diffusion pump and backing pump ; provision being made for ready replacement of the drying agent by arranging a greased cone-joint stopper which when removed permits the withdrawal of the drying agent. The most used substance is phosphorus pentoxide (freedom from arsenic is essential). Various drying agents are listed below, their relative performances can be estimated from the figures (due to Shepherd²) :

Drying agent	Mg. of water per litre of gas at 25° C. after drying
Charcoal trap at — 187° C.	$1 \cdot 6 \cdot 10^{-22}$
Phosphorus pentoxide	$2 \cdot 0 \cdot 10^{-5}$
Magnesium chlorate, $Mg(ClO_3)_2$	$5 \cdot 0 \cdot 10^{-4}$
Melted caustic potash	$2 \cdot 0 \cdot 10^{-3}$
Conc. sulphuric acid	$3 \cdot 0 \cdot 10^{-1}$
Silica gel	$3 \cdot 0 \cdot 10^{-2}$
Calcium oxide	$2 \cdot 0 \cdot 10^{-1}$
Calcium chloride (anhydrous)	$3 \cdot 6 \cdot 10^{-1}$
Zinc chloride	$8 \cdot 0 \cdot 10^{-1}$
Copper sulphate	1·4

¹ See "Zirconium and its Compounds with a High Melting Point", J. D. Fast, *Philips' Tech. Rev.*, Dec., 1938.

² *International Critical Tables*, Vol. III, p. 385.

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The vapour pressure of the drying agent has also to be considered. The above substances have a vapour pressure below 10^{-4} mm. Hg providing they are thoroughly baked or melted before use. Phosphorus pentoxide is most commonly employed. Magnesium chlorate has the advantage that its activity is renewed by baking, but it is considerably less effective than phosphorus pentoxide in practice. Calcium chloride and silica gel are commonly employed inside a vacuum desiccator in which vacuum materials are safely kept before use.

A **Discharge** passed through a vacuum tube by applying some 2000 to 10,000 V. between electrodes sealed into the tube, or formed by the application of a coil or probe carrying high-frequency alternating current (above 100,000 cycles per sec.), when the pressure in

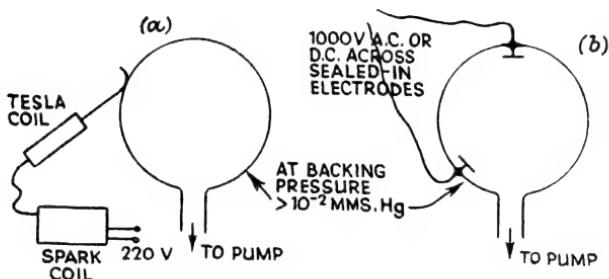


FIG. 95.—Obtaining a Discharge in a Gas.

the tube is between 10^{-1} and 10^{-3} mm. Hg (e.g. before the diffusion pump is switched on) will remove much occluded gas and unimolecular water-vapour films from the vessel surfaces (Fig. 95). This method is very useful when, for practical reasons (e.g. using apparatus with waxed joints), it is impossible to raise the temperature of the walls of the vessel to an appreciable extent.

CHAPTER FIVE

The Applications of High Vacua in Industry

The production of low pressures is becoming, every year, a more and more important factor in industrial progress. Whereas thirty years ago the vacuum was almost exclusively a laboratory phenomenon, the continually improving methods of producing high vacua at speed have resulted in not one, but several new industries. The most important of these is electronics, necessitating the highest order of vacua in sealed vessels so that electron and ion streams can be produced, magnified in number, and diverted without any appreciable undesired ionization because of residual gases.

Exhausting and Activating Radio Valves. The pumping of a modern receiving valve (less than 10 watts anode dissipation)¹ is carried out automatically on a rotating pump system. Thus the so-called twelve-, twenty-four- and sixty-four-head pumps arise whereby a batch of valves are exhausted and activated together. Either the valves are each connected to a separate backing pump and the whole twelve or sixty-four pumps, as the case may be, mounted on a huge turntable, so that, as they rotate, the valves pass automatically through a baking, cathode activation, eddy-current out-gassing, and "gettering" process, or otherwise a batch of some twelve backing pumps in parallel, as a stationary system, is connected to a turntable rotating the valves, and a special oil-sealed, vacuum-tight sliding joint is used.

The valve is pumped through a glass tube sealed by glass-blowing into the "pinch" as illustrated in Fig. 96. This exhaust tube is thrust into a tightly-fitting, thick-walled rubber pressure tubing connecting it to the pump, the inner surface of the rubber is greased with a low vapour-pressure vacuum oil: "Apiezon A" oil or castor-oil are used. A steel screw clamp on this rubber isolates the atmosphere from the pump before the valve is inserted.

The pumping technique aims at removing the maximum amount of gas in the minimum time. All materials used in the valve

¹ The anode dissipation of a valve = anode current \times anode volts under normal operating conditions.

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structure are rendered gas-free before assembly by methods indicated in Chapter Four. A standard procedure is as follows :

Bake the system at 450° C. for 5–10 minutes by allowing valves to pass at a predetermined speed through a long curved oven mounted over the valves during a portion of one complete revolution of the turntable (see Fig. 97). This is, of course, after the backing pump has, for a period of about one minute, exhausted the valve free of air. The baking removes water-vapour.

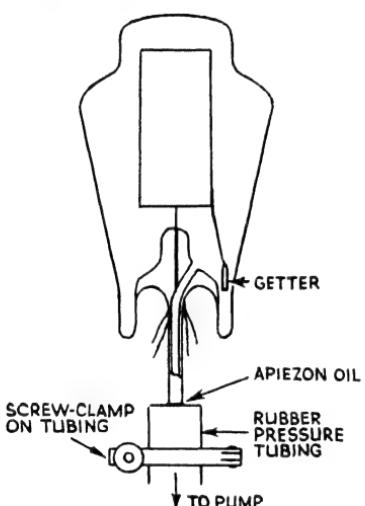


FIG. 96.—Connection of Valve to Pump.

strontium carbonates of the cathode coating are decomposed to oxides, and 10 seconds or so afterwards, the eddy-current heating is resumed whilst the filament or cathode is still hot. The total time of cathode heating is critical and determines the final satisfactory emission of the valve. The valve is readily ruined if the pressure at this stage is too high : the cathode becomes poisoned by the action of residual gases. A satisfactory time, varying from 10 to 100 seconds, depending chiefly on the watts¹ dissipated in the cathode, can only be determined by experiment. The temperature is 1100° C. approx., and, usually, about twice the voltage is applied across valve heater or filament as is later required in normal use. The cathode

Secondly, the valves move to positions on the pump table where eddy-current heater coils automatically fall into position over them, and move with the valves over a short arc of revolution, to heat the metal electrodes to out-gas them. The eddy-current heating time varies between 20 and 90 seconds, depending chiefly on the size of the anode.

After the eddy-current heating, the valve filament or cathode heater, which is wired to an electrical circuit, becomes switched on by the action of the turntable in moving the valve to the appropriate position for electrical contacts to be made. The barium and

¹ Cathode wattage = normal filament or heater volts × heater current.

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and electrodes must be simultaneously heated to avoid interchange of gas between them. Finally, a stage in the revolution of the pump turntable is reached where the valve getter is brought, automatically, opposite an eddy-current heater coil. The action of gettering is carried out at the lowest possible pressure, and the magnesium or barium pellet reduces the pressure in the valve to less than 10^{-5} mm. Hg, even though no diffusion pump is used, in a time not longer than 20 seconds ; then the valve is immediately sealed off the pump. The speed of revolution of the turntable is adjusted to give correct activation times depending on valve size and emission required. A sixty-four-head pump, properly arranged, can produce 300 to 400 valves in one hour.

After sealing from the pump, the valve is put through an activation process to complete the formation of an oxide-coated cathode with satisfactory emission. Current is drawn to the other valve electrodes, which are all strapped together and at a positive potential, for about 2 to 5 minutes with cathode at 900° C. approx. A current of about 100 mA. is caused to pass, depending on the positive potential applied. This current depends largely, however, on the type of valve being activated.

Afterwards the valve is run at normal operating electrode potentials (in a triode the grid is usually earthed) for about half an hour with cathode at 900° C.

This activation process causes an electrolytic action in the cathode coating so that more free barium, which is the active emitter, forms on the cathode surface. Also the electrodes are thoroughly out-gassed by the electron bombardment and any small quantities of gas evolved are absorbed by the barium getter. A cathode of the 8-watt class should be capable of a total emission of 500 mA. if properly activated.

The order of pressure in a valve can be measured by using it like an ionization manometer, i.e. measure the positive ion current to the grid with anode positive and grid negative with respect to the heated cathode. In a small receiver valve this ion current should not exceed one micro-ampere if the vacuum is good.

The annotated diagram, Fig. 97, illustrates the automatic pumping system described above.

Larger valves, with anode dissipations above 10 watts, are pumped in sixes on a six-head stationary pump table. Valves over

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50 watts are usually pumped singly. A transmitter valve with an anode dissipation of 250 watts or more may take several hours to pump. Manifestly the baking and eddy-current heating times must be much increased as the over-all size of the tube increases, and the pumping speed, restricting effect of exhaust tube connected to the valve, selection and out-gassing of the materials in the manufacture of the structure, all become of increasing importance in obtaining an efficient final operation. A much used technique in heating large anodes is to bombard them with electrons from the filament or cathode during pumping. The cathode is overheated by using about twice the usual operating volts across the heater,

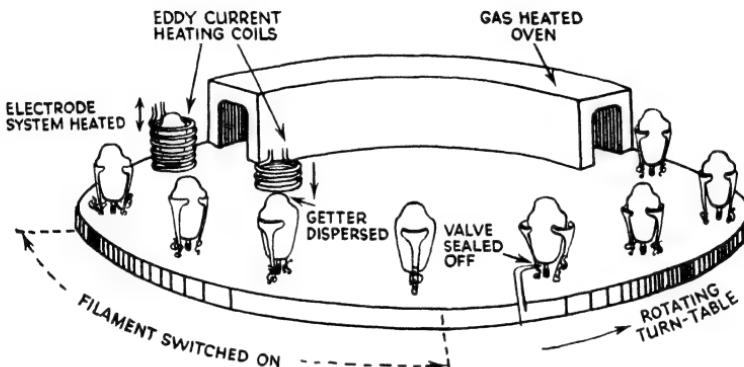


FIG. 97.—Valves on a Rotating Pump Table.

(Valves connected to separate pumps situated below the turntable or via a sliding oil-sealed joint to a bank of pumps.)

and the anode maintained at a positive potential : a large number of electrons hitting the anode at high velocity under the action of the positive field created at the cathode surface will raise the anode temperature to any desired degree without permitting the anode metal to volatilize. The out-gassing action caused by electron bombardment is greater than that caused by raising to the same temperature by eddy-current heating ; apparently the electrons tend to release gas films by their impact as well as by raising the anode temperature.

Cathode-Ray Tubes. The pumping of a hard cathode-ray tube occupies a considerable time and aims at obtaining a vacuum which, over a thousand hours of normal life of the tube, will be

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so good that the electron beam causes negligible ionization of the residual gas. A pressure of 10^{-6} mm. Hg is necessary since the positive ions, in even a small amount, produced by a concentrated electron beam travelling through a distance of several centimetres, will return to the cathode and bombard from its surface the active barium. In the factory as many as six or a dozen tubes may be simultaneously pumped and baked. A single 12"-face tube requires a backing and diffusion pump combination capable of a speed of 20 litres/sec. at the pump jet, and the connecting tube to the electron tube as short and wide as possible. Mercury diffusion pumps in glass or steel with liquid-air freezing traps backed by a fore-pump of ample size are required. If oil-diffusion pumps are used a cooled baffle is essential, and an oil of vapour pressure less than 10^{-6} mm. Hg, otherwise the activation of the thermionic cathode will be unsatisfactory. Pumping a number of tubes simultaneously will require a correspondingly greater pumping speed. Here a useful combination is a large-diameter diffusion pump with a speed up to 200 litres/sec. backed by a fore-pump of sufficiently large displacement.

Pump the tube¹ first with the backing pump only and then find any air-leaks with a Tesla coil. If these leaks are small and in a strain-free region of resistance glass they can be sealed up *in situ* by glass-blowing. Switch on the diffusion pump. If a number of tubes are being evacuated together, a tap arrangement preventing one tube letting air into its neighbour is necessary. Bake the tube in an oven to a temperature depending on the glass of the envelope (see Chapter Four), and for a time determined by the total surface area of the glass. Continue to bake until a McLeod gauge records a pressure of 10^{-6} mm. Hg, or at most 10^{-5} mm. using an ionization gauge. For a 12" tube this takes one to two hours. After cooling give the tube a cut-off test. During 30 minutes the pressure rise should not be greater than to 10^{-5} mm.

The electron gun is then out-gassed by eddy-current heating at a temperature of 700° to 800° C. (red heat), for 5–10 minutes. (The gun must be vacuum-stoved before mounting in the tube.) The activation of the cathode follows a different procedure to that

¹ The schedule given is representative of that adopted with a single, experimental tube. In modern mass production of cathode-ray tubes a much more rapid series of operations is undertaken.

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adopted in the case of valves. Commence with 2 V. across the cathode heater where the normal running voltage is four, and allow the under-run cathode to out-gas for 5 minutes. Then increase the voltage in steps, a volt at a time, with a three-minute period at each stage, till the final voltage is one and a half times the normal operating voltage, i.e. 6 V. for a 4-V. cathode. The wattage dissipated in the cathode is then approximately twice that used after sealing off. The other electrodes of the gun are now strapped together and a current drawn to them from the cathode. The positive voltage on these electrodes is gradually increased, watching the cathode emission with a series ammeter, until some 50 V. per mm. positive field exists between the cathode and the nearest collecting electrode (the grid or modulator). If the emission does not rise as the collector volts is increased, do not go on increasing this voltage but wait for the emission to come up. The series emission or cathode current meter should record 300 to 500 mA. per square centimetre of cathode surface. Adjust the current to about 100 mA. at the appropriate collector volts and bombard the gun electrodes for about 5 minutes to out-gas them at some 700° C. If the temperature increases beyond this value, or a gas discharge occurs, reduce the collector voltage; no gassing should occur if the pumping speed is sufficient. Switch off the collector voltage, and reduce the cathode to its normal running temperature, and then flash the getter as rapidly as possible; then the tube is immediately sealed off the pumps. The getter should, of course, be pre-heated before the cathode activation is commenced, otherwise it will out-gas when finally deposited. The cathode is then activated after sealing the tube off the pumps in the same way as in the case of valves. It is emphasized that poor cathode emission is usually due to unsatisfactory cathode coating: the cathode should always activate readily and rapidly if the pressure is kept below 10^{-6} mm. Hg. A point to note in pumping cathode-ray tubes is to avoid excessive water-vapour entering the tube during glass-sealing to the pump, or during the sudden expansion of air when the backing pumps are first switched on: water-vapour may spoil the fluorescent screen on the tube face.

Gas-Filled Tubes. Both gas-filled valves and cathode-ray tubes are in use. To fill a tube with pure gas to a particular pressure a double stop-cock arrangement is useful.

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In Fig. 98 the gas reservoir contains spectroscopically pure gas. When turned, the second stop-cock with a hole bored in one side allows a small dose of the gas to be passed into the electron tube. Repeated doses raise the pressure of the pure known gas in the tube

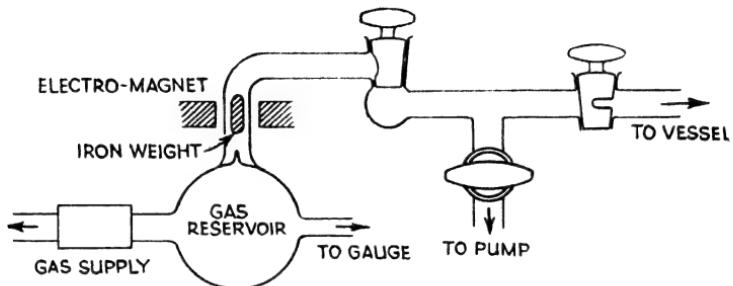


FIG. 98.—Gas-filling a Vessel.

in the desired degree. An ionization or Pirani gauge is the most satisfactory instrument for recording the required pressure.

Fig. 99 shows a suitable arrangement for filling a tube with gas to a higher pressure, recorded by either an oil manometer, suitable

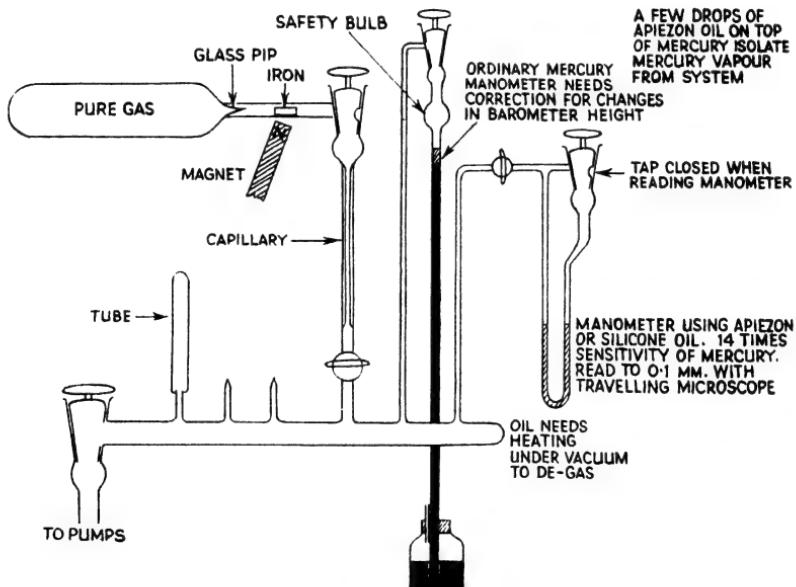


FIG. 99.—Gas-filling a Vessel to Pressures between 1 mm. and 500 mm. Hg.

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for the range from 1 mm. to 10 mm. Hg, or a mercury manometer, useful from 5 mm. to 760 mm. Hg. An optical bellows manometer (page 79) is a convenient device to use in place of the oil manometer, giving greater reliability. Vapours from liquids such as alcohol (used in Geiger counter filling) can be introduced from a small side-vessel with a tap. If a very volatile, obnoxious liquid, such as bromine, is to be introduced in the vapour form, then the side-vessel needs to be placed in carbon dioxide or liquid air whilst the air above the frozen material is pumped away.

A mercury-filled valve is arranged by admitting a small pellet of mercury into the vacuum so that the valve heater causes it to

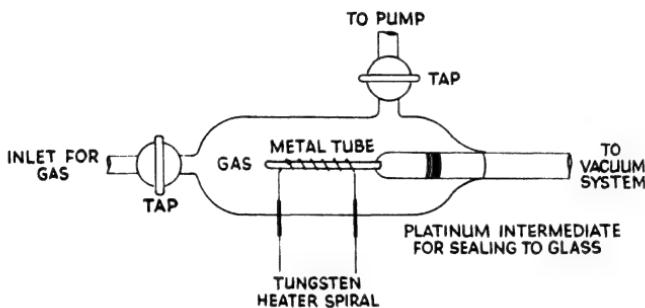


FIG. 100.—Introducing Gas into a System (E. L. Jossem).

vaporize to give the saturation vapour pressure of the mercury at the prevailing temperature.

E. L. Jossem¹ describes a method of introducing gas into a vacuum system which depends on the use of a heated metal or quartz tube as a semi-permeable membrane. A typical piece of apparatus is illustrated in Fig. 100, in which hydrogen is to be introduced into a system through a heated palladium or platinum tube. The gas is let into the bulb by one tap, the other tap provides a means of connecting the bulb to the vacuum pump. When the tungsten spiral wound round the palladium tube is heated by an electric current, then the gas diffuses through this tube into a vacuum system connected at the port indicated. Other gas may be used in place of hydrogen if the palladium is replaced by a suitable metal; thus oxygen diffuses through silver, nickel and copper; nitrogen diffuses through molybdenum, iron and chrom-

¹ E. L. Jossem, *Rev. Sc. Inst.*, **11**, 164, 1940.

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ium; carbon dioxide traverses iron and nickel; whilst a clear fused quartz tube may be used if it is required to introduce helium or other of the rare gases. A constructional detail to note is that, if a metal other than platinum is used, then joining the metal to the glass bulb can be accomplished by an intermediate platinum join, silver-soldered to the metal tube, and fused directly to the glass. If quartz is used, it can be sealed to the glass by means of a graded glass seal. The tube needs to be heated to about 900° C.

A variable capillary gas leak can be conveniently and simply constructed by using a flattened silver tube, according to R. D. Fowler.¹ The operation of this device will be clear on considering



FIG. 101.—A Variable Capillary Gas Leak (R. D. Fowler).

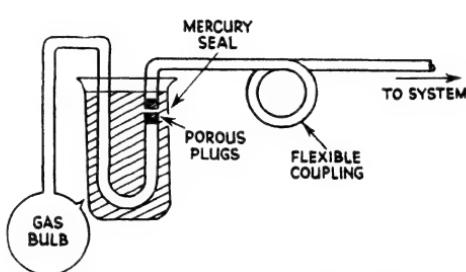


FIG. 102.—A Gas Dosage Device (S. F. Essig).

Fig. 101. It is claimed that the leak rate is readily reproducible in terms of the screw scale reading.

Oxygen from the atmosphere may be introduced into a vacuum chamber by joining a thin-walled silver tubing sealed at one end to the system via a copper-glass seal. On heating the silver tube the atmospheric oxygen diffuses through at a rate fixed by the temperature of the tube and its dimensions.

A further regulation of the pressure in the system can be obtained by heating a tungsten wire in the system itself. At 1600° K. tungsten forms tungsten oxide which evaporates, cleaning up the oxygen in the system.

A gas dosage device introduced by Essig is depicted in Fig. 102. The connecting tube from the gas reservoir to the vacuum system is

¹ R. D. Fowler, *ibid.*, 6, 26, 1935.

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cut across and the open ends are plugged with unglazed porcelain or fired soap-stone which are slightly permeable to gases. Normally these plugs are separated and surrounded by mercury so that no gas can pass the length of the connecting tube. This tubing is, however, wound into a helix over some portion of its length so that it can be flexed readily. Thus, when desired, the two plugs in the mercury well can be brought together by bending the extensible tube, and the gas from the reservoir is permitted to percolate into

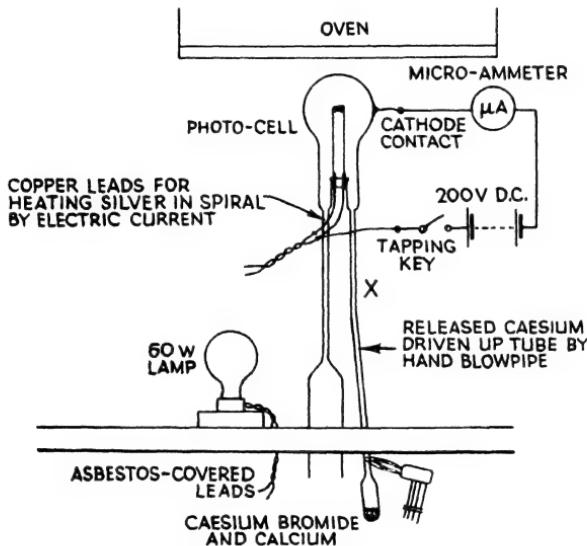


FIG. 103.—Photo-Cell on Pump System.

the system. On allowing the helix to retract, an immediate cut-off of the gas flow is obtained.

The Photo-Cell. The preparation of a vacuum-type photo-cell is a difficult technique in high vacuum work. The commonest type is one with a light sensitive surface of caesium on silver oxide. To prepare a photo-cell in the laboratory a pumping system like that illustrated in Fig. 33 (Chapter One) is suitable. Fig. 103 illustrates the method of setting up the photo-cell on the pump system and the additional apparatus required.

A tube is blown to the shape illustrated in Fig. 104.

The exhaust tube is constricted to facilitate sealing-off from the pump. When the tube is sealed to the pump, as in Fig. 103, a

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long extension tube, some 6 mm. internal diameter, is joined at *X*. The closed end of this tube is arranged well below the asbestos platform. At the bottom of the tube is a mixture of about 10 mgm. of caesium bromide and two or three small chips (2 mm. \times 1 mm. about) of calcium. A glass-blowing joint is made at *X* to seal the caesium tube to the main tube after the latter is sealed to the pump. At the centre of the photo-cell bulb is a tungsten spiral welded to nickel support wire from the pinch. This tungsten spiral is 0.1 mm.

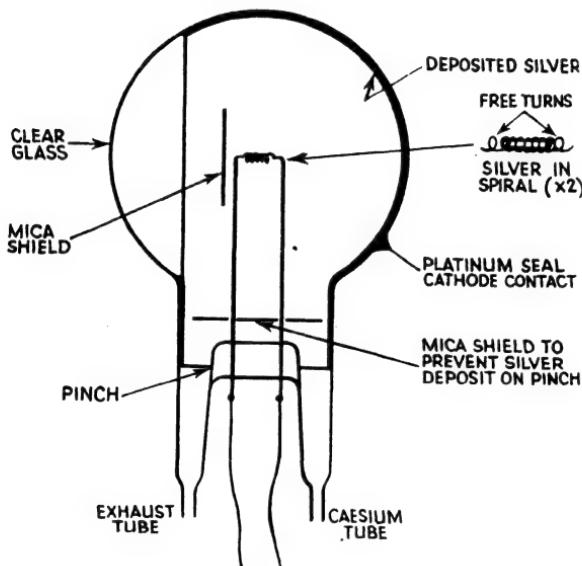


FIG. 104.—A Photo-Cell.

diameter and some eight turns about a piece of pure silver wire 2 mm. long and 1 mm. in diameter, the spiral projecting by one clear turn on either side of silver wire (see subsidiary sketch, Fig. 104). Copper-clad leads through the pinch enable an electric current to be passed through the spiral. All glass used must be previously thoroughly cleansed (chromic acid and distilled-water washings) and dried. The photo-cell bulb is evacuated to 10^{-6} mm. Hg with a backing and diffusion pump combination using a liquid-air trap. A pumping speed of 4 litres/sec. at 10^{-4} mm. Hg is adequate. After leaks have been found and sealed up, the tube is baked for 20 minutes at a temperature depending on the glass of the bulb

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(400° C. for soda-glass ; 550° C. for Pyrex glass). Before this bake it is advisable to heat the cæsium tube till some cæsium is released into the bulb so as to "cæsiate" the glass, otherwise the affinity of the glass for cæsium during the true photo-activation process may ruin the results obtained.

After the tube is cool and a cut-off test has been passed the activation is begun. The spiral containing silver is first pre-heated below the temperature at which evaporation of silver commences, so as to de-gas the spiral thoroughly. At a yellow heat, passing 1 to 2 amps. from a 6-V. accumulator, the silver will commence to evaporate and quickly form a near-opaque layer on the glass walls.

A nickel or mica shield round the spiral will throw a shadow within which no silver is deposited, so that a clear-glass window is left in the envelope to admit the light-beam. A short out-gassing bake at about 160° C., with the pressure still below 10^{-6} mm. Hg recorded by a McLeod or ionization gauge, will ensure that succeeding activation stages can be commenced. The photo-tube is cut off from the pumps by a greased stop-cock or mercury cut-off and then a side-tube which contains potassium chlorate or barium peroxide is heated by a gas-air flame so that oxygen is evolved. A pressure rise to some 10^{-2} mm. Hg is permitted.

A probe electrode connected to the output of a 50-watt high-frequency oscillator operating at about 500 kilocycles will, when passed over the outside of the photo-tube, ionize the oxygen in the tube to give a yellowish-green discharge. The silver surface will become oxidized, a change which can be followed through successive surface colour changes. The degree of oxidation is found only by experience based on previous experimental work : it is a vital factor in determining the tube's final photosensitivity. Care must be taken, and practice is needed, to achieve a uniformity of oxidation over the surface area of the photo-cathode : a well-diffused discharge will be necessary for this result.

The oxygen is pumped away and the tube baked at 150° C. (the AgO is affected above 180° C.) to remove final traces of gas. Then cæsium is sublimed in small doses up the side-tube by heating the glass tube with an air-gas flame, the doses occupying about one-inch length of the cæsium tube as a surface film on its walls. The cæsiation is best done in one stage, but experimenting is neces-

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sary, doing the processing in gradual steps, to ascertain by experience how much caesium is required for a particular tube. Between each admission of caesium the tube must be baked at 150°–170° C. till a maximum photosensitivity is recorded. The variations of sensitivity can be followed by putting a 60-watt lamp inside the oven, and measuring on a series microammeter the photo-emission when 200 V. is applied between the cathode and anode using the circuit of Fig. 105. The caesiation is continued till photo-emission is constant, and then the tube is sealed from the pump. Too much caesium will ruin the emission. Only by several months' preparatory experimental work can first-class results be obtained.

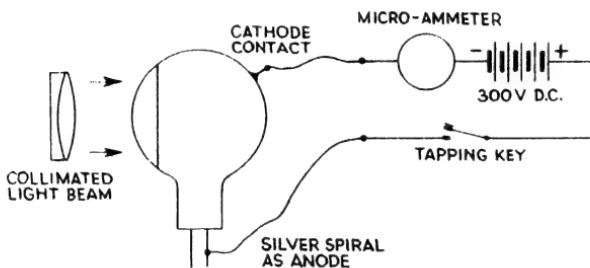


FIG. 105.—Testing the Emission from a Photo-Cell.

In mass-producing photo-cells the above tedious laboratory process is replaced by a method whereby a predetermined weighed amount of caesium is admitted. Many methods and patents have been recorded for achieving constant, predictable oxidation and caesiation conditions of a photo-cell made according to a fixed schedule. The caesium is usually released in photo-cell mass production by eddy-current heating a metal disc on which is mounted a pellet containing caesium chromate, chromic oxide and powdered aluminium, in proportions about 1 : 6 : 2·5.

Many types of photo-cell have been produced, each one having various techniques associated with its production. Photo-cells may be made on cathodes based on glass, mica, silver, gold and inert metals. Semi-transparent silver layers have been photo-activated so that light falling on one side of the layer will cause an electron emission from the other side. Various metals other than caesium have been used to get particular spectral sensitivities. The following

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is a record of photo-sensitivities of various cells, using a tungsten lamp source at 2800° C.

Material of photo-cathode	Wave-length at which response is maximum	Micro-amps. per milliwatt at max.
A.U.S		
Cs-O	3500 and 8000	3·0 and 3·2
Cs-Mg. . . .	3300	3·2
K-H	4500	5·3
K-O	3300	3·7

Other cells used are sodium (white-light sensitivity 0·5 μ A/lumen); potassium (1·0 μ A/lumen); rubidium (0·44 μ A/lumen, green sensitive); sodium on sulphur (7·0 μ A/lumen); Cs on CsO on Ag (up to 50 or 60 μ A/lumen).

The Deposition of Metal Films in Vacuum. The production of a hard, adhesive, metallic coating on a surface support is of some industrial importance. Layers of aluminium and silver on glass to form front-surface reflecting mirrors, coatings of silver on mica in the manufacture of radio-receiver condensers, and the deposition of thin uniform gold films for filter purposes, are a few applications.

One of the recent advances in the optical industry has been the aluminizing of optically worked mirrors for telescopes, searchlights, gauge projectors, etc.

Aluminium is a metal with a high reflecting power (ref. Chapter Six), adheres well to glass, and has a surface film of transparent aluminium oxide which is a valuable protection against the chemical action of weathering.

Aluminized mirrors have been made by evaporation of the metal *in vacuo* up to sizes 200" in diameter.¹ The technique is described below for aluminizing mirrors up to about 24" in diameter using a metal vacuum vessel. The reader will readily adapt his ideas to suit smaller or larger mirrors, in glass or other chambers, remembering that the larger the vessels the greater the pumping speed of the system used should be if it is to produce an adequately low pressure during the evaporation process. Thus a chamber 14" in diameter and 10" high needs a pumping speed of 20 litres/sec. at 10⁻³ mm. Hg. Even greater pumping speeds, up to 200 litres/sec., are desir-

¹ J. Strong, *Astrophys. Jour.*, 83, 401, 1936.

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able in mass production methods where the vessel cannot be continually cleaned of old films.

A vessel for larger mirrors, say 40" in diameter, requires a number of pumps in parallel, or one of the pumps capable of a speed above 200 litres/sec. A diffusion pump and backing pump must be used. A backing pump only will enable a film to be obtained in a small vessel, but it will not be satisfactory over a long period of use. Liquid air is not necessary if "Apiezon A" oil, or one

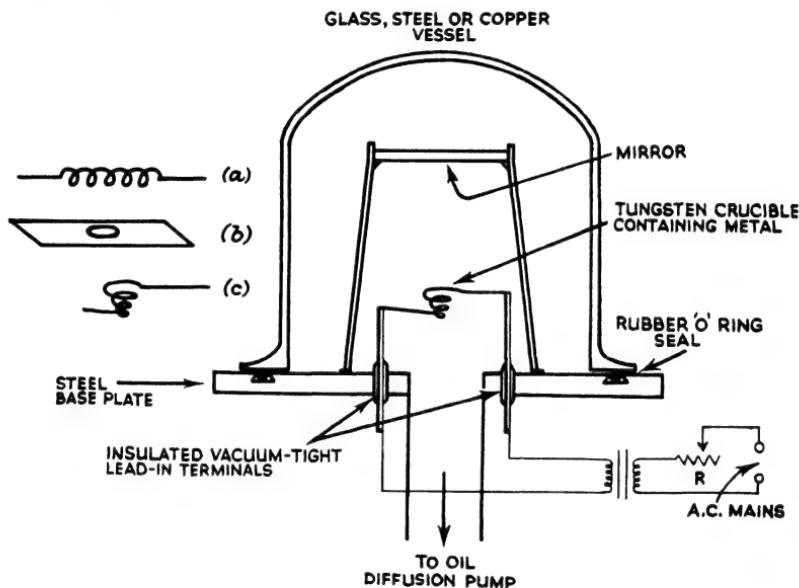


FIG. 106.—Diagrammatic Drawing of a Simple Plant for the Deposition of a Metal Film.

Three types of filament employed are shown : tungsten wire of about 0.5 mm. dia. is used for forms (a) and (c); molybdenum strip is suitable for (b).

of the other vacuum oils for diffusion pumps, is employed. The vapour pressure of the oil must be below 10^{-5} mm. Hg. If a mercury pump is used then liquid air is essential, as mercury vapour is ruinous to aluminium; carbon dioxide snow ($-78^{\circ}\text{C}.$) is not satisfactory as a freezing-trap compound for this work, as water-vapour tends to evaporate from the upper, cooler parts of the trap and brown films result.

Suitable gears are illustrated in Figs. 106, 107 and 108.

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To obtain a satisfactory deposit cleanliness and low pressure are essential. First wash the glass on which a film is required with a soap solution, then rinse in water, and soak in chromic acid. Such "wet" cleaning should be continued until a water-film spreads uniformly over the glass surface indicating freedom from grease. Caustic soda should be used with caution as it will etch glass if

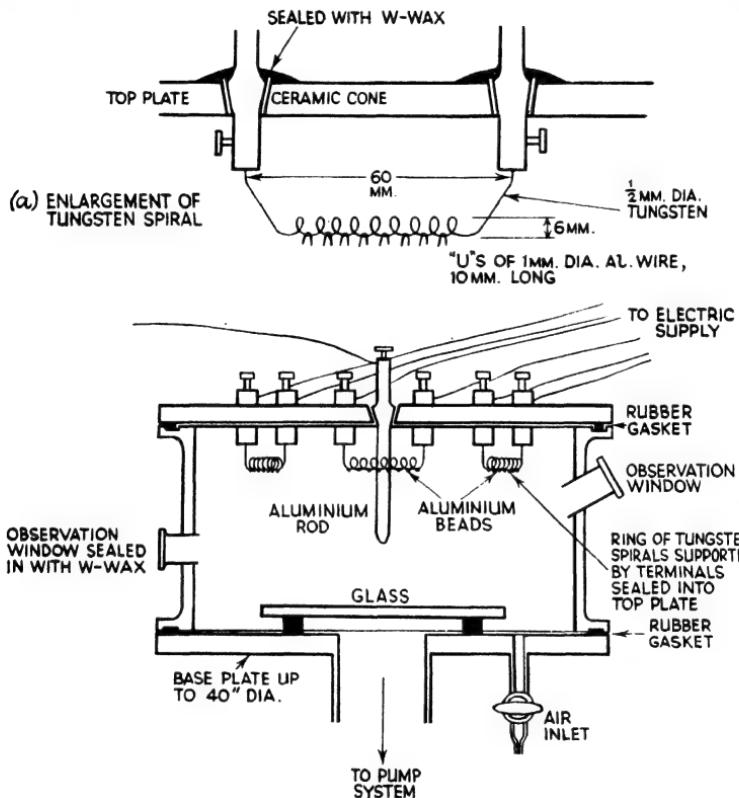


FIG. 107.—Volatilizing Aluminium in Vacuum.

sufficiently concentrated. Dry the mirror with cotton-wool, and then polish with cotton-wool and alcohol until a uniform water-vapour film is formed on its surface when breathed upon. Dirt which is very difficult to remove may be released by the judicious application of a light rouge pad.

Before inserting the mirror in the metallizing chamber, tungsten

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spirals supporting aluminium must be made and formed *in vacuo* (J. Strong, *Astrophys. Jour.*, June, 1936). About a foot of 0·5-mm. diameter clean tungsten wire is wound into an open spiral and little "U"s of 1-mm. diameter aluminium wire placed over the loops and squeezed on with tweezers. See Fig. 107a. These tungsten spirals are set up between terminals in the chamber and are

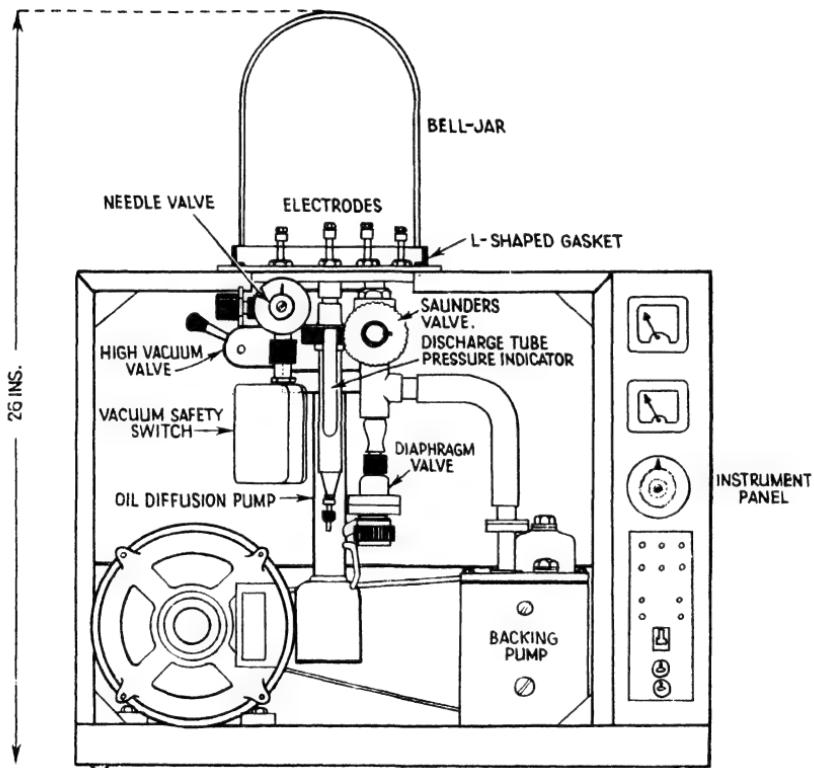


FIG. 108.—A Small, Commercial Evaporating Unit (W. Edwards and Co., Ltd.)

capable of being heated by the passage of electric current from a step-down A.C. mains transformer with a series rheostat. The chamber is pumped to less than 10^{-4} mm. Hg as registered by a "black-out" on a discharge tube with 15 kV. applied, or by a McLeod or Philips gauge. The spirals are then heated to about 800° - 1000° C. so that, under the action of the surface-tension of the

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molten aluminium, beads of this metal are formed. This forming of the spiral must be done before the glass is inserted, or a removable metal shield, operated from a vacuum-tight greased cone, or a Wilson seal, may be used to prevent the metal depositing on the glass.

Insert the glass to be coated and re-evacuate the vessel with the backing pump. Before switching on the diffusion pumps, apply up to 15 KV. A.C. or D.C. between the metal base-plate and the insulated electrode, so as to bombard the glass surface with ions for some 15 minutes. This will remove thin water-films and grease-films on the glass. Alternatively, or in addition, an electric heater is arranged behind the glass plate in the vacuum so as to roast the glass to about 300° C. before deposition of the metal is begun.

This bombardment must be done before forming the beads if a moving shield arrangement is used as suggested above, i.e. bead formation and mirror deposition done during the same evacuation. After an ion bombardment cleaning, pump as rapidly as possible with the diffusion pumps till a pressure less than 10^{-4} mm. Hg is obtained and then begin to heat the tungsten spirals one by one for about 30 seconds each, and repeatedly, at 1400° C. (a yellow heat) to allow a bright aluminium film to be deposited. After the film has formed, allow apparatus to cool down, cut off the pump and admit air. Leave the mirror a whole day before use to allow the protective oxide film to form on the aluminium surface. If the film is brown or dark, then water-vapour or too high a pressure must have been present during the deposition. Likewise, if bubbles or blisters appear in the film after standing in air, the cause is invariably too poor a pressure during deposition. This may be due to too high an initial pressure, or too low a pumping speed whilst spirals are hot. Films that do not strongly adhere to the glass are due to bad cleaning of the glass, bad vacuum or inadequate rate of deposition. A stronger film can be made by depositing a base layer of chromium on the glass just before evaporating the aluminium. Two sets of spirals must therefore be set up inside the metallizing chamber, because it is useless to allow air to the chromium film before depositing the aluminium.

Metal films of desired light transmission are best made by passing a parallel beam of light through the glass during the deposition and recording initial and final transmitted light intensities by a photo-cell.

Many metals may be deposited using the above technique. Heater

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spirals or boats of tungsten or molybdenum will serve. To evaporate gold or silver, the metal may be in wire form and the tungsten wire wound round it. To deposit nickel, chromium, platinum or rhodium it is best to electro-plate the metal on the clean tungsten heater, otherwise it will be difficult to attain a sufficiently high temperature. Chromium chips or nickel wire placed inside a tungsten spiral of 0·5 mm. wire will, however, evaporate at a glowing white heat. Hot aluminium and beryllium dissolve tungsten, necessitating the special spiral of thick wire due to J. Strong described above.

Rhodium films on glass are of great value as robust mirrors : the electro-plated spiral method has been successfully applied.

The Heraeus Co. in Germany have patented a method whereby strips of rhodium, of much larger surface area than a wire, are heated to just below the melting-point of rhodium by the passage of an electric current of several amps. The low rate of deposition from 1 sq. cm. of metal at a temperature below its melting-point is counteracted by the use of a large surface area.

Platinum is a metal which it is very difficult to evaporate as it tends to form into one drop on the heater wire and cause it to burn out. It is best deposited by sputtering (*see* below).

H. M. O'Bryan¹ introduced a technique for evaporating metals in vacuum whereby they were placed in a cup-shaped graphite crucible surrounded by a tungsten heater wire. Fig. 109 illustrates the method. The tungsten spiral is heated to 2300° C. so as to emit electrons which then bombard the crucible which is maintained 1000 V. positive with respect to the spiral. The high-velocity electron bombardment heats the crucible, and the metal inside it, to the evaporation temperature.

This technique enables very high temperatures to be attained, but it is beset with difficulties. To avoid an ion discharge under the action of the volts between crucible and spiral a pressure below 10^{-5} mm. Hg must be continuously maintained, and this is inhibited by the out-gassing of the hot carbon which re-absorbs gas every time the vessel is opened to air. A long pre-heating before raising the temperature sufficiently to cause evaporation avoids this snag to some extent.

The evaporation temperature of various metals is given in the

¹ H. M. O'Bryan, *Rev. Sc. Inst.*, 5, 1934.

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following table, together with notes on difficulties encountered in the technique.

Metal	Temperature ° C.	Remarks
Cæsium	433	Readily evaporated
Rubidium	450	" "
Potassium	480	" "
Cadmium	541	" "
Sodium	565	" "
Zinc.	623	Tungsten heater or molybdenum boat used, readily evaporated
Magnesium	712	" "
Strontium	811	" "
Lithium	821	" "
Calcium	878	" "
Barium	905	" "
Bismuth	913	" "
Antimony	973	" "
Lead	1000	" "
Tin	1148	" "
Chromium	1190	Difficult as does not liquefy ; plated spiral used
Silver	1319	Readily evaporated
Gold	1445	" "
Aluminium.	1461	" "
Copper	1542	" "
Iron.	1694	" "
Nickel	1717	" "
Platinum	2332	Very difficult
Molybdenum	2755	Very difficult, only thin films in long period
Tungsten	3505	" "

Many non-metallic materials may be evaporated in vacuum to form thin films on a support. Three recent applications have been, firstly, the evaporating of silica as a protective film on weatherable glass, and as a thin insulator dielectric¹; secondly, the evaporation of fluorite and other materials of low refractive index on glass to increase the light transmission²; thirdly, the manufacture of inter-

¹ G. Hass and N. Scott (*J.O.S.A.*, **39**, 179, 1949) describe the use of silicon monoxide, which evaporates at lower temperature, as a protective coating for lenses and mirrors. It oxidizes to the dioxide on exposure of film to air. Evaporated from tantalum boat, 4" × 14" × 0.008" using current of 250 A.

² K. Blodgett, *Phys. Rev.*, **55**, 391, 1939.

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ference filters in optics.¹ These materials, and several other salts not affected by temperature, can be evaporated by placing small pellets or crystals in a tungsten spiral or in a molybdenum boat and heating as for metals. Silica rod of 1 mm. diameter inside a closely-fitting tungsten or tantalum spiral of 0·5-mm. diameter wire will evaporate slowly. The films obtained are tinged faintly with brown tungsten oxide if evaporated from tungsten. A small tantalum crucible holding fused silica and heated by electron bombardment

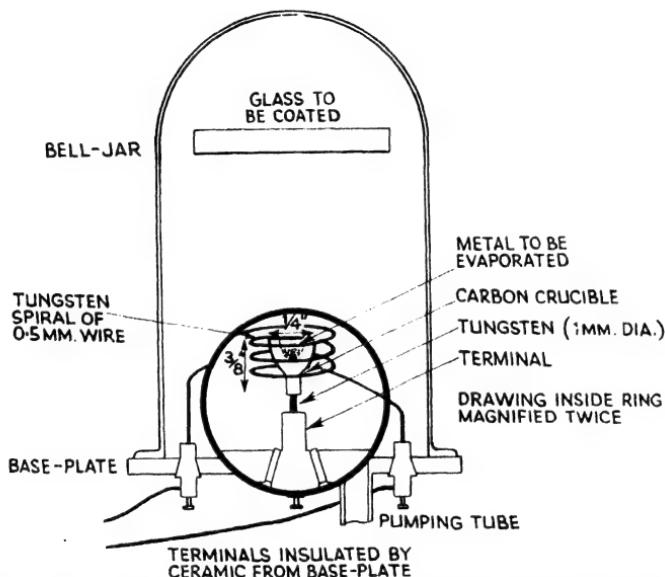


FIG. 109.—Volatileizing a Metal by Electron Bombardment Heating.

from a heated tungsten spiral is an excellent way to obtain pure thin silica films. This compares with O'Bryan's technique mentioned above, but tantalum gives less difficulty than carbon.

To evaporate fluorite or calcium fluoride, it is best to press the powder into a small cylindrical pellet which is then placed inside a spiral of about 1·5 mm. diameter of 0·3 mm. tungsten. Crystals tend to spit off small particles when evaporated in vacuum, ruining the smooth finish of the coating.

Fluorite crystals, and other crystalline substances, may also be

¹ K. M. Greenland, *Endeavour*, 11, 143, 1952.

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evaporated by fusing them into a small pellet which is then placed in a crucible-form tungsten spiral or on a molybdenum boat. Calcium fluoride acts chemically on tungsten so that it is best to platinum or rhodium-plate the spiral to be used. Crucibles, formed by painting a water suspension of alumina or beryllium oxide on to a closely wound tungsten spiral, have been applied by Olsen, Smith and Crittenden¹ to the evaporation of metals and refractories at temperatures up to 1,600° C. Before use, these crucibles are dried and sintered at about 1,300° C.

The physical properties of crystals and even of metals are radically changed when evaporated to form a thin film in vacuum. Any crystalline structure is altered or destroyed because the refractory vapour in vacuum is in the form of separate atoms or molecules which, when they deposit on the support, rarely rearrange to form the structure present in the normal form. Thus the fluorescent and photo-conductive properties of crystals are lost when so evaporated. The resistivity of metals in the very thin film form is abnormally high because of the greater spaces separating aggregates of atoms.

Cathodic Sputtering. A method of obtaining thin films of metals whereby a sheet of the metal is bombarded with the positive ions in a gas discharge so that atoms are released. A convenient "sputtering" gear is shown in Fig. 110.

The chamber is first evacuated by a backing and diffusion pump combination to a pressure less than 10^{-5} mm. Hg as recorded by a McLeod gauge. Then a slow trickle of gas (usually hydrogen or argon) is admitted to the chamber through a needle-valve. The rate of inflow of the gas is balanced against the rate of pumping so that a suitable equilibrium pressure is attained. Between the plate *A* of the metal to be sputtered (e.g. silver), and the base-plate *B*, a potential of 1000 to 2000 V. is applied with the plate *A* negative, i.e. the metal is the cathode. An alternating potential, effective during negative half-cycles, may be used and readily obtained from the A.C. mains using a step-up transformer. The pressure of gas in the chamber is varied by the needle-valve until the glow discharge excited between the cathode and anode has a dark space extending for 1 cm. approximately beneath the cathode. The positive ions of gas produced in the discharge will then impinge on the negative cathode under the action of the large fall of potential

¹ Olsen, Smith and Crittenden, *Jour. Appld. Phys.*, **16**, 425, 1945.

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in the dark space, and their momentum will be sufficient to dislodge aggregates of atoms from the cathode, so that the glass beneath the cathode becomes covered with a film of the cathode metal.

The support for the glass (or other substance on which a film is required) is best made of aluminium which does not sputter appreciably. The glass shield *S*, and glass tube round lead-in

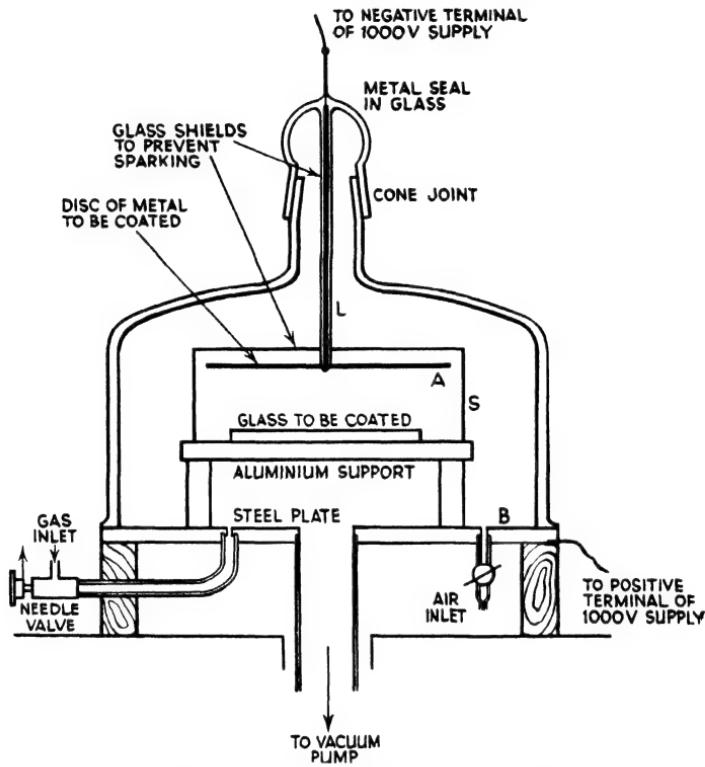


FIG. 110.—A "Sputtering" Apparatus.

wire *L* to the cathode, are necessary to avoid a discharge forming outside the effective region between the cathode and glass which is to receive the deposited film. Using platinum, in a hydrogen atmosphere and 1000 V. between electrodes about 4 cm. apart, an opaque film will be obtained in 50 to 60 minutes.

The rate of sputtering increases with the ion current density, and with the cathode fall in potential, and depends on the molecular

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weight of the positive ions. Hydrogen behaves exceptionally, giving a much faster rate of sputtering compared with other gases than would be expected from relative molecular weights : hydrogen is much used in sputtering. Some gas is absorbed during the action so that the needle-valve leak rate may have to be regulated.

Günterschulze¹ gives following relative rates of sputtering of the various metals in a hydrogen atmosphere where the cathode fall of potential is 770 V. and the current density 7 mA./sq. cm.; the number quoted against the metal is the rate of sputtering in milligrams per ampere-hour : Bi 1470 ; Te 1200 ; As 1100 ; Tl 1080 ; Sb 890 ; Ag 740 ; Au 460 ; Pb 400 ; Zn 340 ; Cu 300 ; C 262 ; Sn 196 ; Fe 68 ; Ni 65 ; W 57 ; Co 56 ; Mo 56 ; Mn 38 ; Cd 32 ; Al 29 ; Cr 27 ; Ta 16 ; Mg 9.

For practical purposes aluminium, chromium and magnesium do not sputter. A film of aluminium can be sputtered in argon gas, but it will not be as optically bright as an evaporated film. Air can be used for sputtering : in fact, a backing pump pressure of about 10^{-1} mm. Hg forms a useful "sputtering" atmosphere without the necessity for specially introducing gas. However, metals which oxidize at all readily are not suitably sputtered in air. Argon gas gives the most rapid rate of sputtering, but is much more expensive than hydrogen.

The rate of sputtering of metals in descending order in an argon atmosphere is : Cd ; Ag ; Pb ; Au ; Sb ; Sn ; Bi ; Cu ; Pt ; Ni ; Fe ; W ; Zn ; Si ; Al ; Mg. Generally the evaporation process is much more satisfactory than the "sputtering" process. The notable exception is in the case of platinum where it is much easier to obtain a film by "sputtering". Rhodium can be sputtered at a rate about one-quarter of that for platinum, but the films obtained are not so durable or bright as those obtained by the evaporation technique. Sputtered films contain much more occluded gas than evaporated films.

¹ Günterschulze, *Zeits. für Physik.*, **36**, 563, 1926.

CHAPTER SIX

The Properties of Materials Important in High Vacuum Technique

GASES

Gas	Density in grm./ litre at N.T.P.	Viscosity 10^{-5} poise ¹	Critical pressure atmos.	Critical temp. °C.	B. Pt. °C.
Air	1.2928	18.1	—	—	—
Ammonia	0.7708	10.8	112.3	132.9	33.5
Argon	1.783	21.0	48.0	122.44	186
Bromine	7.139	—	131.0	302	58.8
Carbon monoxide	1.2502	18.4	35.0	141	190
Carbon dioxide	1.9768	16.0	72.7	31.1	78.5
Chlorine	3.220	14.7	76.1	144.0	34.0
Helium	0.1785	18.9	2.26	267.91	269
Hydrogen	0.0899	9.5	12.8	239.91	253
Krypton	3.68	23.3	54.24	62.6	156
Neon	0.900	29.8	26.86	228.71	246
Nitrogen	1.251	18.4	33.49	147.13	196
Oxygen	1.4290	20.9	49.71	118.82	183
Water-vapour	0.606	9.8	58.22	374.0	—
Xenon	5.85	21.1	57.2	14.7	108

Gas	Mean molecular diameter ² 10^{-8} cm.	Mean free path ² 10^{-6} cm.	First excitation potential volts (for atom)	First ionization potential volts (for atom)
Air	—	6.69	—	—
Argon	3.67	6.67	11.6	15.8
Bromine	—	—	—	11.85
Carbon monoxide	3.4	—	—	—
Carbon dioxide	4.65	4.40	—	—
Chlorine	4.96	4.57	—	13.01
Helium	2.2	19.36	19.7	24.6
Hydrogen	2.75	12.26	10.16	13.59
Krypton	4.15	5.34	—	14.0
Mercury-vapour	5.10	8.3	4.7	10.4
Neon	2.6	13.75	16.6	21.5
Nitrogen	3.75	6.7	10.6	14.5
Oxygen	3.64	7.10	9.4	13.6
Water-vapour	4.7	4.5	—	—
Xenon	4.9	3.93	8.3	12.1

¹ At 760 mm. Hg and 20° C.

² At 760 mm. Hg and 25° C.

THE ELEMENTS

Element	$M_F \cdot C_F$	$B_F \cdot C_F$	Density, g./cm. ³	Atomic weight	Expansion °C ²⁰ /10° C.	Thermal cond., sec. cm. ² C ⁻¹ at 20° C.	Resistivity millimeter at 20° C.	Temp. of fusion or melting at 20° C.	Specific heat, g./gm. at 20° C.
Aluminum.	630	1645	2.70	26.97	26	5040	3.21	0.0036	0.2103
Antimony.	—	1630	4.86	539.94	5	420	0.0038	—	—
Argon.	—	—	1.150	3.75	—	—	—	0.08	—
Barium.	1280	1500	1.84	1.84	12	—	—	0.07	0.425
Bismuth.	1560	1560	9.78	9.02	190	—	0.004	0.307	0.939
Boron.	2700	772	58.8	58.8	12	—	—	0.055	—
Bromine.	73.3	670	7.72	7.72	30	220	7.54	0.055	0.048
Cadmium.	32.5	132	1.87	1.87	49.08	—	—	0.15	—
Cesium.	28.5	130	1.32	1.32	7.9	—	—	0.16	—
Calcium.	3500	3930	Graphite	2.22	12.0	Diamond	10.5	0.003	—
Cerium.	81.5	1400	6.80	140.13	—	—	—	0.045	—
Chlorine.	—	102	—	3.34	Table 1	135.46	—	0.226	—
Chromium.	1830	2200	6.92	52.0	—	—	—	0.110	—
Cobalt.	1860	3460	8.89	58.94	12	920	13.1	0.104	—
Fluorine.	—	2223	3.00	8.89	—	—	971	0.004	0.023
Gallium.	30.2	23.00	1.87	5.93	—	—	172	—	—
Germanium.	938	2160	5.46	69.72	—	—	—	—	—
Helium.	1.272	—	2.68	1.272	14	7000	2.42	0.0036	0.074
Hydrogen.	—	252.7	—	252.7	Table 1	14.0002	—	0.031	—
Iodium.	1.55	1.84	7.28	11.48	—	—	—	0.057	—
Iron.	152.7	323.5	2.42	19.692	46	—	—	0.054	—
Krypton.	—	1.57	2.42	19.692	97	—	—	0.113	—
Lanthanum.	832	327	1.755	1.34	12	1610	5.3	0.006	—
Lead.	—	—	—	—	—	12.0	—	0.045	—
Lithium.	186	120	7.41	5.54	—	—	—	0.039	—
Manganese.	1230	1900	3.57	13.56	21	3460	9.77	0.0009	0.033
Molybdenum.	2622	3600	10.16	96.0	5	48	0.0047	0.072	—
Neon.	84.9	—	—	—	—	—	—	—	—
Nickel.	1452	3000	—	—	Table 1	120.18	—	—	—
Niobium.	1950	—	8.5	58.69	13	1420	1.18	0.0027	0.109
Oxygen.	2760	0	195.8	92.91	—	—	—	0.028	—
Palladium.	1.555	2.540	—	19.6008	7	—	—	0.031	—
Phosphorus.	1744	2779	1.83	1.0600	—	—	—	0.059	—
Potassium.	63.3	3760	2.05	106.7	12.2	1680	10.7	0.0037	0.1922
Praseodymium.	940	—	0.86	31.02	124	—	—	0.19	—
Radium.	9600	—	6.48	139.10	80	2300	10.5	—	—
Ruthenium.	38.9	—	—	140.92	—	—	—	—	—
Samarium.	1900	—	12.3	225.97	9	2100	6.0	0.058	—
Selenium.	1350	—	1.53	182.44	90	—	—	0.08	—
Silver.	227	690	7.75	150.43	10	—	—	0.061	—
Sodium.	960.5	2152	10.55	150.43	37	—	—	0.074	—
Tellurium.	977.7	1366	0.97	107.88	19	9740	1.62	0.0036	0.056
Tantalum.	112.8	46.6	2.6	23.00	70	3100	4 $\times 10^{10}$	0.0033	0.283
Thallium.	295.9	451	1.63	87.06	70	1300	5	0.036	—
Titanium.	1302	1457	6.25	181.45	—	—	21.0	0.048	—
Tin.	1.232	2270	1.86	127.5	17	900	31	0.033	—
Tungsten.	1800	4830	4.5	264.39	23	1550	40	0.0044	0.154
Vanadium.	3380	—	19.3	184.0	23	—	11.4	0.0044	—
Xenon.	1620	—	6.0	50.94	4	3500	5.5	0.0052	0.034
Yttrium.	111.8	109	—	131.93	—	—	—	0.015	—
Zinc.	4.924	918	7.1	88.92	28	2650	5.92	0.0035	0.0974
	1857	—	6.4	91.22	—	—	—	—	0.067

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Note :

Mean Free Path for Nitrogen at 0° C.

mm. Hg	cm.
760	6.7×10^{-6}
1	5.1×10^{-3}
10^{-3}	5.1

and follows law that $L \propto \frac{1}{\text{pressure}}$, where L = mean free path.

For other gases given in table above compare with nitrogen. The mean molecular diameters are average values of results of several investigators.

*Conditions in gaseous discharge*¹:

Gas	Cathode dark space width at pressure = 1 mm. Hg		Cathode potential drop	
	Al cathode	Fe cathode	Al cathode	Fe cathode
Argon	cm. 0.29	cm. 0.36	volts 100	volts 165
Helium	1.32	1.66	141	153
Hydrogen	0.72	0.90	171	250
Mercury vapour	0.33	0.34	245	298
Neon	0.64	0.72	120	150
Nitrogen	0.31	0.42	179	215
Oxygen	0.24	0.31	311	290

Emission Spectra of Gases. Lines at following wave-lengths in A.U.S² (10^{-8} cm.):

Argon—4159, 4192, 4198, 4201, 4259, 4300, 4334, 4703, 5452, 5607, 5912, 6031, 6059, extending from violet to orange region of visible spectrum.

Carbon dioxide—3590, 3884, 4123, 4216, 4393, 4511, 4735, 4835, 5165, 5198, 5610, 6079 (from violet to orange).

Helium—3188, 3889, 4026, 4471, 4713, 4921, 5015, 5875, 6678, 7065.

¹ Espe and Knoll, *Werkstoffkunde der Hochvakuumtechnik*, Julius Springer.

² A.U. = Ångstrom unit = 10^{-8} cm.

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Hydrogen—3750, 3771, 3798, 3836, 3889, 3970, 4102, 4340, 4861, 6563 and others in extreme ultra-violet from 1030 to 1675.

Neon—5853, 5882, 5945, 5976, 6030, 6075, 6096, 6129, 6143, 6164, 6182, 6217, 6267, 6305, 6383, 6402, 6507 (from yellow to red, red is pronounced).

Nitrogen—Band spectrum edges at 4648, 4666, 4723, 4813, 5340, 5614, 5755, 5804, 5854, 5906, 5959, 6013, 6069 (blue to orange).

Oxygen—3919, 3973, 4070, 4072, 4076, 4415, 5208 and other band and continuous spectra.

The Diffusion of Gases Through Metals. The rate at which a gas diffuses through a metal is of obvious interest to the vacuum worker, but it is not proposed to enter into a discussion of the theory of this diffusion here, but simply to gather together some useful practical information on the diffusion of pure gases into a vacuum. A detailed exposition of this subject will be found in the book by Smithells,¹ together with much information on the diffusion of mixtures of gases, effects at high pressure, etc.

Fick gives a diffusion law indicating that the rate of diffusion is inversely proportional to the thickness of the metal at constant pressure and temperature. This law has been proved experimentally. The rate of diffusion of a gas through a metal is usually quoted as D , being the volume of gas in c.c. at N.T.P. diffusing per second through 1 sq. cm. of surface 1 mm. thick.

The diffusion rate increases with temperature rise, the equation $D = ae^{-q/T}$ representing results within the limit of experimental error, a and q being constants depending on the gas and metal concerned, e is the exponential base 2.71828, T is the absolute temperature, the pressure being kept constant.

As the gas pressure difference across the metal interface increases the rate of diffusion increases. Borelius and Lindblom² propose an equation $D = k(\sqrt{p} - \sqrt{p_t})$ for this effect, where p is the pressure, and p_t is a threshold pressure below which diffusion does not occur. Smithells and Ransley,³ however, do not support this contention about a threshold pressure, but consider that, although a \sqrt{p} law obtains, yet diffusion persists down to pressures as low as

¹ C. J. Smithells, *Gases and Metals*, Chapman and Hall Ltd., 1937.

² Borelius and Lindblom, *Ann. der Phys.*, **82**, 201, 1927.

³ Smithells and Ransley, *Proc. Roy. Soc.*, **150**, 172, 1935.

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0.1 mm. Hg, the deviation from Borelius and Lindblom's results depending on θ , the fraction of the surface covered by the absorbed gas, according to the equation $D = k\theta\sqrt{p}$.

Some typical results for various cases are :

(a) Hydrogen through Copper.

Pressure 129 mm.	Temp.	300° C.	400° C.	450° C.
	D	0.2×10^{-7}	10^{-7}	3×10^{-7}

(b) Hydrogen through Iron.¹

	$\sqrt{\text{Pressure}} =$	2 mm.	5 mm.	10 mm.	15 mm.	20 mm.
At 700° C.	$D =$	0	4.5×10^{-6}	12×10^{-6}	17×10^{-6}	24×10^{-6}
532° C.	$D =$	0	2×10^{-6}	3.5×10^{-6}	6×10^{-6}	8×10^{-6}
300° C.	$D =$	0	0.5×10^{-6}	0.1×10^{-6}	0.2×10^{-6}	0.6×10^{-6}

(c) Hydrogen through Nickel.

Pressure 760 mm.	Temp.	450° C.	600° C.	700° C.
	D	0.2×10^{-4}	0.8×10^{-4}	2.2×10^{-4}

(d) Hydrogen through Copper.²

	$\sqrt{\text{Pressure}} =$	0 mm.	2 mm.	4 mm.	6 mm.	8 mm.	10 mm.
At 450° C.	$D =$	0	2×10^{-8}	6×10^{-8}	13×10^{-8}	18×10^{-8}	25×10^{-8}

(e) Hydrogen through Nickel.

	$\sqrt{\text{Pressure}} =$	0 mm.	5 mm.	10 mm.	15 mm.	20 mm.
At 522° C.	$D =$	0	0.3×10^{-6}	1.4×10^{-6}	2.5×10^{-6}	3.5×10^{-6}
426° C.	$D =$	0	0.1×10^{-6}	0.4×10^{-6}	0.8×10^{-6}	1.2×10^{-6}

¹ Borelius and Lindblom, *loc. cit.*

² Smithells and Ransley, *loc. cit.*

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(f) Hydrogen through Palladium.¹

	$\sqrt{\text{Pressure}} =$	0 mm.	5 mm.	10 mm.	15 mm.
At 508° C.	$D =$	0	0.7×10^{-8}	2×10^{-8}	3.6×10^{-8}

(g) Oxygen through Silver.

	$\sqrt{\text{Pressure}} =$	0 mm.	5 mm.	10 mm.	15 mm.
At 400° C.	$D =$	0	10^{-8}	3.5×10^{-8}	7×10^{-8}

(h) Hydrogen through Aluminium.

	$\sqrt{\text{Pressure}} =$	0 mm.	5 mm.	10 mm.	15 mm.
At 558° C.	$D =$	0	0.17×10^{-7}	0.35×10^{-7}	0.53×10^{-7}

These results, due to various observers, must be taken to apply in the case of pure gases in the molecular form, where the state of purity of the metals is of a high order. The effect of impurities in the gas or metal are, in some cases, considerable. Gases in the nascent state, e.g. electrolytically produced at the seat of diffusion, will have far higher diffusion rates. The state of the surface of the metal, e.g. whether polished, machined, or roughened, also has a considerable influence on the adsorption of gas, and hence on its diffusion through the metal. In addition the history of the metal and its heat treatment decides its crystalline condition, which also influences diffusivity.

¹ Lombard, *Comptes Rendus*, 177, 116, 1923.

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THE VAPOUR PRESSURES OF METALS AND CARBON AT VARIOUS TEMPERATURES ¹

Vapour Pressures above 1 mm. Hg

Metal	Vapour pressure is						
	1 mm. at °C.	5 mm. at °C.	10 mm. at °C.	20 mm. at °C.	100 mm. at °C.	200 mm. at °C.	760 mm. at °C.
Aluminium . .	1284	1421	1487	1555	1749	1844	2056
Antimony . .	886	984	1033	1084	1223	1288	1440
Barium . .	860	984	1049	1120	1301	1403	1638
Beryllium . .	1582						
Bismuth . .	1021	1099	1136	1177	1271	1319	1420
Cadmium . .	394	455	484	516	611	658	765
Caesium . .	279	341	375	409	509	561	690
Calcium . .	817	926	983	1046	1207	1288	1487
Carbon . .	3586	3828	3946	4069	4373	4516	4827
Chromium . .	1616	1768	1845	1928	2139	2243	2482
Cobalt . .	2056						
Copper . .	1628	1795	1879	1970	2207	2325	2595
Gold . .	1869	2059	2154	2256	2521	2657	2966
Iron . .	1787	1957	2039	2128	2360	2475	2735
Lead . .	973	1099	1162	1234	1421	1519	1744
Lithium . .	723	828	881	940	1097	1178	1372
Magnesium . .	621	702	743	789	909	967	1107
Manganese . .	1292	1434	1505	1583	1792	1900	2151
Molybdenum . .	3102	3393	3535	3690	4109	4322	4804
Nickel . .	1810	1979	2057	2143	2364	2473	2732
Palladium . .	2000						
Platinum . .	2730	3007	3146	3302	3714	3923	4407
Potassium . .	341	408	443	483	586	643	774
Rubidium . .	297	358	389	422	514	563	679
Silver . .	1357	1500	1575	1658	1865	1971	2212
Sodium . .	439	511	549	589	701	758	892
Strontium . .	750	847	898	953	1111	1192	1384
Tantalum . .	> 3000						
Thorium . .	2715						
Tin . .	1492	1634	1703	1777	1968	2063	2270
Tungsten . .	3990	4337	4507	4690	5168	5483	5927
Uranium . .	487	558	593	632	736	788	907
Zinc . .	450						

¹ See D. R. Stull, *Ind. Eng. Chem.*, **39**, 517, 1947. See also S. Dushman, *Scientific Foundations of Vacuum Technique* (Chapman and Hall Ltd., 1949), pp. 746 et seq.

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Vapour Pressures below 1 mm. Hg

Metal	Vapour pressure is				
	10 ⁻⁵ mm. at °C.	10 ⁻⁴ mm. at °C.	10 ⁻³ mm. at °C.	10 ⁻² mm. at °C.	10 ⁻¹ mm. at °C.
Aluminium . . .	724	808	889	996	1123
Antimony . . .	466	525	595	678	779
Barium . . .	418	476	546	629	730
Beryllium . . .	942	1029	1130	1246	1395
Bismuth . . .	474	536	609	698	802
Cadmium . . .	148	180	220	264	321
Cæsium . . .	45	74	110	153	207
Calcium . . .	408	463	528	605	700
Carbon . . .	2129	2288	2471	2681	2926
Chromium . . .	907	992	1090	1205	1342
Cobalt . . .	1249	1362	1494	1649	1833
Copper . . .	946	1035	1141	1273	1432
Gold . . .	1083	1190	1316	1465	1646
Iron . . .	1094	1195	1310	1447	1602
Lead . . .	483	548	625	718	832
Lithium . . .	325	377	439	514	607
Magnesium . . .	287	331	383	443	515
Manganese . . .	717	791	878	980	1103
Molybdenum . . .	1923	2095	2295	2533	—
Nickel . . .	1157	1257	1371	1510	1679
Palladium . . .	1156	1271	1405	1566	1759
Platinum . . .	1606	1744	1904	2090	2313
Potassium . . .	91	123	161	207	265
Rubidium . . .	60	89	123	165	217
Silver . . .	767	848	936	1047	1184
Sodium . . .	158	195	238	291	356
Strontium . . .	361	413	475	549	639
Tantalum . . .	2407	2599	2820	—	—
Thorium . . .	1686	1831	1999	2196	2431
Tin . . .	823	922	1042	1189	1373
Tungsten . . .	2554	2767	3016	3309	—
Uranium . . .	1461	1585	1730	1898	2098
Zinc . . .	211	248	292	343	405
Zirconium . . .	1527	1660	1816	2001	2212

The values for vapour pressures given in these two tables must be regarded as approximate only, despite the fact that three or four significant figures are quoted. Many of the values are calculated from thermodynamic formulæ and the application of kinetic theory to results in the rate of evaporation of metals. There are considerable disparities between the results quoted by various workers.

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THE PROPERTIES AND USES OF SUBSTANCES EMPLOYED IN HIGH VACUUM TECHNIQUE IN ALPHABETICAL ORDER

Aluminium. Reflectivity of evaporated aluminium film on glass.

λ in A.U.S	Percentage normal incident light reflected
White light	88
2,500	80
4,000	90
6,000	90
8,000	85
10,000	90
12,000	85

- Uses :* (1) Preparing aluminized mirrors giving high reflecting power, especially high relative to other metals in U.V. region.
(2) As a getter material : little used nowadays.
(3) Drawn and cast aluminium readily vacuum-tight. Used for vacuum chambers (e.g. in metallizing).
(4) As an electrode in a gas discharge tube has advantage that does not sputter. However, its low melting-point prohibits its use in most tubes. Also difficult to out-gas thoroughly, especially of hydrogen.
(5) Used as Lenard window in X-ray tubes as particularly transparent to X-radiation.

A thin film of oxide (Al_2O_3) forms on a fresh surface of aluminium as soon as exposed to the atmosphere, and increases in thickness for some 60 days. Often "anodizing" used to increase this oxide film thickness for artificially hardening aluminium surfaces.

Aluminium may be soldered to other metals if it is first copper-plated. Soldering can also be accomplished using ultrasonic methods. Aluminium is much used for the manufacture of the jet systems of oil diffusion pumps because it possesses the advantages of lightness, inertness to hot oils, and high thermal conductivity.

PROPERTIES OF MATERIALS

Apiezon products : *see under Cements.*

Asbestos

Specific gravity	= 2.5 approx.
Melting point	= 1150° C. for Horneblende asbestos
	= 1550° C. for Serpentine asbestos
Thermal conductivity	= 3.5×10^{-4} cal.cm. $^{-1}$ sec. $^{-1}$ °C. $^{-1}$

The commonest heat insulator. Used in oven manufacture and for lagging. Obtainable as asbestos string, sheet, paper, and wool. Also available under various commercial names as mixtures with cements, and as slate asbestos to form a harder, more robust compound.

Bakelite. Resistivity = 2×10^{16} ohm-cm.

A valuable electrical insulator : readily worked. Used for switch panels, valve bases and holders. Its use is deprecated inside a vacuum as an electric insulator because vapour pressure is high when temperature increased above 30° C. Chars at 280° C. Does not absorb water.

Bakelite cements are used for fixing valve caps to tubes. Melts at 80° C. Soluble in methylated spirits.

Barium

- Uses :* (1) The most used getter material.
(2) Much employed (mixed with strontium) as cathode material, giving a copious thermionic emission.

For a standard barium oxide, strontium oxide cathode coating at—

Temp.	Electron emission mA./sq. cm.
477° C.	0
527° C.	50
977° C.	750
997° C.	2000

Photo-electric work function = 1.7 V.

Beeswax. A mixture formed by melting together equal parts of beeswax and resin is a useful vacuum wax. It softens at 50° C.

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and melts at 60° C. If applied hot round a demountable joint (e.g. a bell-jar flange to flat metal) it forms a gas-tight seal adhering well to glasses and metals. Is used for fixing glass to tool in optical working. Soluble in carbon tetrachloride and alcohol.

Beryllium. Used like aluminium for Lenard windows in X-ray apparatus. Gives a film when evaporated on to glass which reflects strongly in ultra-violet region.

This metal has great hardness and is comparatively easily alloyed with most of the heavy metals, giving hard, corrosion-resisting alloys.

Beryllium-copper and beryllium-nickel are two examples which have been especially developed, the range of alloys ordinarily used containing 2·75% Be. By adding up to 0·5% cobalt or nickel more uniform properties are produced after hardening. Beryllium-copper, in particular, is used in all types of springs, electrical contacts, diaphragms, bellows, Bourdon gauge tubes, etc. A low-temperature heat treatment is used to harden the finished product. Resistance to fatigue is greater than for spring steel, and the life of the alloys is some twenty times that for phosphor-bronze. The thermal and electrical conductivity for beryllium-copper are 20% of those for copper. It can be obtained in sheets up to 6" wide, and down to 0·004" thick.

Soft soldering to beryllium alloys is satisfactory if it is done after the final heat treatment, but otherwise silver solder or brazing is necessary, since the temperature for heat treatment is greater than the temperature at which soft solder flows. Zinc chloride or resin are satisfactory fluxes for soft soldering.

Machining is readily carried out, but for drawing, pressing or bending the metal must be softened by quenching.

Brass. A copper-zinc alloy. Density = 8·5. Spec. ht. = 0·09 cal./gm./° C. Resistivity = 8×10^{-6} ohm-cm. Temp. coefficient of resistivity = 0·0015.

Uses: Extensively employed in construction of all types of scientific apparatus. Percentage of zinc varies. Tombac is a tubing of 28% zinc with corrugated walls which is flexible. Available in various diameters it is useful for high-vacuum connexions using metal apparatus. Withstands temperatures up to 450° C.

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Whereas brass tubing is with difficulty made vacuum-tight, tombac is non-porous at normal temperatures.

Bronze is a copper-tin alloy : useful for castings in large vacuum apparatus. Is prone to air-leaks which can only be avoided by heavily painting casting surface, or by a coating of W-wax. Not so porous as brass.

Cadmium. A metal used for special photo-cell cathodes, and as a cadmium lamp in which a discharge through hot cadmium vapour gives a reddish glow. Used as a standard of wave-length in spectroscopy. Reference line is red cadmium at 6438·4696 A.U.S.

A cadmium photo-cell formed by evaporating cadmium on to a glass support *in vacuo* is sensitive to light of wave-length less than 3200 A.U.S.

T. Wilner¹ describes the use of a cadmium trap for mercury vapour in the line between diffusion pump and vessel. Cadmium is heated to 300° to 400° C., but regions on either side of container are water cooled.

Cæsium. An alkali metal which oxidizes very rapidly at atmospheric pressure. Prepared in vacuum by heating a mixture of caesium bromide or chloride with calcium.

Photo-emission and thermal emission both high. Principal application is as an evaporated layer on an oxidized silver layer *in vacuo* to form a photo-sensitive surface. (See Chapter Five.)

Photo-electric work function = 1·36 V. A pure caesium layer gives photo-electrons for incident light from $\lambda = 4200$ to 6400 A.U.S with a maximum at 5600 A.U.S.

For a Ag-O-Cs cell λ in A.U.S	Emission in μA . per milliwatt of incident energy
3,000	1·5
4,000	2·5
6,000	1·5
8,000	3·0
10,000	0·5

Calcium. An alkali-earth metal : should be kept in non-oxidizing atmosphere or liquid.

¹ T. Wilner, *Rev. Sc. Inst.*, **20**, 527, 1949.

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Calcium is used for releasing caesium from the halide in photo-cell manufacture. Used also as a getter when a non-conductive, almost invisible deposit is required in a valve where very low "noise" required. Combines with all but rare, inert gases. Was used in early thermionic cathodes, but not so effective as barium. Photo-electric work function = 2.4 V.

Carbon. Specific gravity = 2.22 (graphite).
= 3.51 (diamond).

Resistivity at 20° C.

for gas carbon = 0.004 to 0.007 ohm-cm.

for graphite = 0.003 ohm-cm.

for diamond = 10^{12} ohm-cm.

Sublimes when heated.

Porosity = 25–30%.

Uses: Coco-nut shell charcoal as a gas absorber, and as graphite for manufacture of small transmitting valve anodes. Difficult to out-gas by eddy-current heating.

Gas	Vol. at N.T.P. of absorbed gas per c.c. of charcoal at	
	0° C.	— 187° C. (Liquid air)
Oxygen	c.c. 18	c.c. 230
Nitrogen	15	155
Hydrogen	4	135
Helium	2	15

Carbon in the form of colloidal graphite suspension in water or oil is called " aquadag ". It is widely used in vacuum tubes. It bakes on to glass at 450° C. whereas for coating metal it is best sprayed on from a gun and vacuum-baked at 900° C. Greatest adhesion is obtained by coating the graphite on to a metal which has the same coefficient of linear expansion. Some typical applications are as electrical screening on the walls of a tube, for making contact to deposited metal coatings, to reduce secondary, back and photo-emission on valve electrodes, as a coating on cooling fins, or on the anode of a valve to act as an efficient black-body heat radiator.

PROPERTIES OF MATERIALS

It absorbs caesium strongly. Can be used to improve the electrical conductivity of paper, asbestos, etc., by impregnation technique.

Cements. Included are details of most materials used for sealing cones or flanges of a demountable joint together, or for covering a small hole or crack in a vacuum vessel. The use of one or other material depends primarily on the temperature it will have to withstand in use.

Apiezon Compounds—supplied by Shell Chemicals Ltd.

“*Apiezon L*” is a grease supplied in metal tube containers. In use, a thin film of the grease is worked between well-fitting cone or flange joints till no “air-lines” remain. Vapour pressure = 5×10^{-8} mm. Hg at 20° C. “*Apiezon M*” and “*N*” are similar, rather more viscous greases, more suitable if room temperature is above 20° C. If “*Apiezon M*” grease is baked in vacuum at 90° C. for two hours its vapour pressure can be reduced to 10^{-8} mm. Hg. These greases must be kept free from contact with air when not in use: they give more trouble due to occluded air than to high vapour pressure. Suitable for use in glass and metal stop-cocks. A stop-cock will give continual use up to three months if properly greased.¹

“*Apiezon Q*” compound is a plasticine-like substance consisting of graphite mixed with the low vapour-pressure residues of paraffin oil distillation products. It is a semi-solid compound which can be readily pressed with the fingers round a joint in glass or metal to render it vacuum-tight.

Vapour pressure at 20° C. = 10^{-4} mm. Hg. Becomes too liquid for use at temperatures above 30° C.

“*Apiezon W*” wax has a very low vapour pressure ($< 10^{-8}$ mm. Hg at 20° C.). It is a hard substance, supplied in sticks, very useful for semi-demountable joints in glass or metal where fusing or welding is impossible because of high temperatures necessary. It is applied to the joint at 100° C. It is best to heat the surface as well as the wax if a fairly permanent, gas-tight joint is required.

¹ Shell Chemicals Ltd. state that the vapour pressure of the more expensive “L” grease is 10^{-3} mm. Hg at 300° C., implying a vapour pressure of 10^{-11} mm. at 20° C. The “M” grease is said to have a vapour pressure of 10^{-6} to 10^{-7} mm. at 50° C.

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not liable to develop cracks. Softening point = 60°–70° C. Soluble in xylene.

“*Apiezon W40*” and “*W 100*” waxes have softening points of 30° C. and 50° C. respectively. They are useful in place of the ordinary “W” wax where application to a joint which cannot be heated much is required. Vapour pressures are 10⁻³ mm. Hg at 180° C.

A cement known as “*Araldite*” is marketed by Aero Research Ltd. It is available in two forms : (i) powder, (ii) sticks, and in either a light brown colour or “silver”. This cement is useful for a vacuum joint between two flat or well-fitting surfaces. For bonding pieces of glass together, and as an adhesive for light alloys, it is particularly valuable. It possesses high adhesion to glass, porcelain, ceramics, metals and mica. After surfaces are cleaned, the powder is sprinkled over them, and they are then permanently bonded by the application of heat alone, without pressure. Suggested curing times and temperatures are : 240° C. for 10 minutes ; 220° C. for 20 minutes ; 190° C. for 1 hour and 180° C. for 2 hours. If “stick” form “*Araldite*” is used, the surfaces to be joined must first be heated to 120° C., the rod rubbed on, the surfaces joined, and a curing operation, as for powder form, undertaken.

Dow Corning Silicone Greases, which are remarkably stable from –40° C. to 200° C. with constant viscosity characteristics, are available in two forms : (i) D.C. stop-cock grease, for general laboratory use, (ii) D.C. high vacuum grease, suitable for operating at pressures below 10⁻⁶ mm. Hg. They are rather more viscous than the Apiezon greases at normal room temperatures, but function very well, especially if the stop-cock is liable to temperature extremes in use.

Edwards W.E. wax 3 and *W.E.6* are shellac-based, brown waxes which, unlike “W” wax and Picein, are soluble in alcohol but fairly insoluble in aromatic hydrocarbons like benzene, toluene, etc. Softening point of “*W.E.3*” is 80° C., and of “*W.E.6*” it is 90° C.

Glyptal is a viscous, condensation resin which resists the action of water, acids and alkalis. It dries to a hard glossy film in 8 hours at 70° F. Soluble in acetone. Xylol used as thinner. Valuable for painting on to regions of porosity in walls of vacuum systems and vessels.

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Khotinsky Cement is a mixture of shellac and pitch obtained from Caroline tar. Softens at 50° C. Comparatively insoluble in usual organic liquids and common acids. Useful for joints at temperatures below 40° C. Vapour pressure at 20° C. = 10^{-3} mm. Hg approx.

Picein—a hard black wax of vapour pressure = 10^{-8} mm. Hg at 20° C., and 3–10 mm. at 50° C. Softens at 50° C. Chemically inert to usual organic liquids and inorganic acids. Used similarly to “W” wax.

Plaster of Paris—used for cementing caps to valves, usually of large transmitting types. Setting is speeded up by admixture with salt.

Sealing Wax—soluble in alcohol, is convenient in solution for painting on a leaky joint where the temperature cannot be increased to apply “W” wax.

Shellac—soluble in alcohol and butyl phthalate, applicable to leaks in same way as sealing wax, but has the advantage that, dissolved in butyl phthalate, gives less likelihood of high vapour pressures.

Silver Chloride applied at 450° C. is a useful sealing cement which will withstand high temperatures. Can also be used for sealing small flat windows on glass vessels, seals into discharge tubes, etc. Soluble in sodium thiosulphate.

Ceramics¹ are being used to an increasing extent as insulating materials in vacuum tubes. As a means of rigid support of electrodes they are ideally clean and readily gas-free. Particularly useful in transmitting valves. Low-loss valve bases for use at very high frequencies are best made of ceramics such as glazed porcelain. Aluminium oxide and magnesium oxide are used as thin tubes or as sprayed on insulator in a suspension of amyl acetate to cover the thin tungsten heater wires in indirectly heated cathodes. Ceramics moulded to particular shapes are commercially available. Lathe-working and drilling are difficult owing to the hard, brittle nature of these materials.² Porcelain tubes can be sealed to glass by glass-blowing technique.

¹ See Data Book, Ceramics, 1938–9.

² An exception is the valuable pyrophyllite which is an excellent insulator, and readily machined before firing.

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Properties of the commoner ceramics:

Material	Comp.	Fusion pt. °C.	Soft pt. under load	Conductivity cal.cm. ⁻¹ sec. ⁻¹ °C. ⁻¹	Resistivity ohm-cm.
Silica brick . . .	95% SiO ₂	1720	1620	0.0026 (700° C.) 0.0043 (1000°)	9700 (1300° C.) 22 (1550°)
Magnesite . . .	MgO	2000	1600	—	1550 (30°)
Clay brick . . .	Al ₂ O ₃ ; SiO ₂	1680	1300	0.0042 (1000°)	1300 (1300°) 60 (1400°)
Magnesia-spinel . .	MgO; Al ₂ O ₃	2130	2000	—	—
Sillimanite . . .	Al ₂ O ₃ ; SiO ₂	1810	1700	0.0044	—
Zirconium oxide . .	Al ₂ O ₃ ; SiO ₂	2550	1850	—	—
Alundum . . .	Al ₂ O ₃	2000	1750	0.0083	75 × 10 ⁴ (1040°) 48 × 10 ⁴ (530°)
Chromite . . .	Cr ₂ O ₃ ; FeO	2050	1510	0.0034	2800 (800°) 390 (1400°)
Porcelain . . .	—	1700	1500	—	5 × 10 ⁸ (400°) 10 ¹⁵ (20°) 10 ⁸ (400°)
Steatite . . .	—	—	—	—	—

Colombium (or Niobium): *see Tantalum metals.*

Constantan: *see Copper.*

Copper is used whenever good heat and electrical conductivity are of great advantage. Examples are copper in oil diffusion pumps, copper grid support wires in valves, copper valve anodes.

Copper can be sealed to glass. Housekeeper¹ has sealed glass tubes to copper tubes of 4" diameter; the end of the copper tube being bevelled to a "feather" edge. The cooled anode type of valve made in this way can dissipate up to 10 kW.

A nickel-iron alloy wire thinly coated with copper can be readily sealed into most glasses to give a vacuum-tight lead-in wire. Such wires are used as pinch-wires in the usual receiver valve. The wire is usually treated with a thin layer of borax flux to facilitate sealing to glass. The name "red-platinum" is given to these wires.

A combination of 72% copper and 28% zinc called "Tombac" used for flexible corrugated wall tubing (*see Brass*). Constantan is an alloy of 45% nickel with 55% copper having practically zero temperature coefficient. Eureka is almost identical with constantan.

Safe currents for copper are 270 amps./cm.² for No. 12 wire (diam. = 2.64 mm.); 430 amps./cm.² for No. 22 wire (diam. = 0.711 mm.); 500 amps./cm.² for smaller diameters. A piece of No. 38 copper wire makes a 5-amp. fuse.

Copper vacuum vessels are comparatively non-porous, and can

¹ Housekeeper, *Amer. Inst. Elec. Eng.*, **42**, 954, 1923.

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be conveniently used for large-scale work. Tinning or silvering the copper surface is recommended because pure copper corrodes and oxidizes readily in the atmosphere.

Ebonite is a useful, readily-worked insulating material suitable for switch panels, valve-sockets, etc. It is not much used *in vacuo* because of high vapour pressure, and inability to withstand temperature increases above 40° C. As a small insulator in a continuously pumped system it will serve, however, if kept cool.

Resistivity = 2·10¹⁵ ohm-cm.

Dielectric constant = 2·7-2·9.

Fernico is a metal which seals to glass. It has similar properties to Kovar. (See Kovar.)

Glasses.¹ A great variety of glasses exists, but only a few of them are generally encountered in high-vacuum work, the others are mostly of interest in optics. The commonest are soda glass, lead glass and the borosilicate glasses like "Pyrex" and "Hysil": not a very precise classification, however, because the properties of glass vary, depending on the manufacturer. Thus German and English soda glasses differ considerably in their physical properties.

The simplest glass is 60%-75% silicon dioxide (SiO_2) in the form of quartz sand with 5%-15% alkali oxide and 5%-15% alkali-earth oxide. The commonest acidic oxides used are silicon dioxide and boric oxide (B_2O_3); basic oxides employed include lime (CaO), potash (K_2O), soda-lime (Na_2O), magnesia (MgO) and oxides of barium, zinc, manganese, lead, aluminium, iron, arsenic, lithium, tin and zirconium. The elements fluorine and selenium also occur. For colouring glasses, oxides of metals are used: such metals are iron (glass blue-green to yellow-green), cobalt (blue), nickel (colour variable depending on percentage nickel oxide), chromium (green), copper (blue to green), iridium (grey to black), cadmium (yellow), uranium (yellow—fluoresces markedly), titanium (yellow-gold).

Window glass, plate glass and bottle glass are mixtures of silica, soda and lime to which alumina is sometimes added to improve the durability of the glass, and diminish the risk of devitrification during manufacture.

¹ The writer is indebted to Chance Bros. and Co., Ltd., for information on glass.

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The percentages of constituents of common glasses are :

Type	Name and reference number	SiO ₂	Na ₂ O	K ₂ O	CaO	BaO	MgO	PbO	B ₂ O ₃	Al ₂ O ₃	As ₂ O ₃	Sb ₂ O ₃
Soda-lime	Wembley X8	69	16.2	0.7	6.1	—	3.2	—	1	3.5	—	—
	Chance GW1	75	16	—	7	—	—	—	—	1	—	1
	Plate glass	73	13	—	13	—	—	—	—	1	—	1
Lead	Wembley L1	56.5	5.1	7.2	—	—	—	30.2	0.2	0.8	—	—
	Chance GW2	60	8	5	—	—	—	26	—	0.1	—	0.5
	Lead crystal	56	—	12	—	—	—	31.8	—	0.2	—	—
Hard	Pyrex	80.2	3.9	0.3	0.2	—	—	—	12	3	0.3	—
	Hysil GHI	80.4	4.2	—	0.3	—	—	—	12.4	2.7	—	—
	C9 (tungsten sealing)	75	3.4	1.9	0.3	—	—	—	18.3	1.1	—	—
	Intasil GS1 (tungsten sealing)	74.5	5	—	0.5	3	—	—	13	4	—	—
	Chance GS3 (Kovar sealing)	67	3	4.6	—	—	—	—	21	4	—	0.4
	Wembley HH (molybdenum)	73	2.4	4.2	3	—	—	—	14.4	3	—	—
	Chance GS4 (molybdenum)	76	6	—	1	3	—	—	10	4	—	—

The mixture of glass constituents is heated in a furnace during manufacture to 1400°–1500° C. till it is free of bubbles and then poured into moulds or drawn into sheet glass or tubing. The subsequent cooling of the glass must be carefully regulated, an annealing schedule being followed depending on the nature and mass of the glass. Such annealing must always be done after glass has been in the molten state, e.g. after glass-blowing. Pyrex, Hysil and Monax glass tubing can be worked in the form of thin wall tubing without careful subsequent annealing, whereas the glass-blower usually cools soda glass very carefully down to a luminous flame temperature, finally depositing a protective carbon deposit on the cooling glass from the coal gas flame. If a complicated piece of glass apparatus, or pressed glass as in a valve pinch, is made then it must be annealed in an oven. The procedure adopted in the case of freshly manufactured glass is to pass it slowly through a fire-brick tunnel or "lehr" so that the temperature gradient during cooling is within specified limits. During cooling the viscosity is usually too high and the rate of heat diffusion too low to allow crystal growth. At a temperature depending on the glass it is endothermic, and then a sudden change in physical properties

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occurs. Some glasses are prone to devitrification, or the formation of small crystals ; usually such crystals are of silica in the case of the borosilicate glasses, and of calcium silicate in soda-lime glasses.

This devitrification is not easily avoided by modifying the annealing process ; a well-prepared batch of constituents is arranged so that devitrification does not occur. Devitrification is particularly likely to occur if the glass is cooled too slowly, or maintained at the softening temperature for a considerable time. Other defects occurring in manufacture include stones, or particles of the constituents in the glass, striæ which are veins of glass of different refractive index, bubbles and seeds (fine bubbles). Colouring due to iron content in the sand used is difficult to avoid.

The strain in glass resulting from imperfect annealing is vividly demonstrated by a strain-viewer. A polarized light beam from a pile of plates or a sheet of " polaroid " is passed through the glass and viewed by a Nicol prism or polaroid eye-piece. Strain in the glass gives a coloured effect in the field of view, clearly de-marking the region of strain. A quarter-wave plate inserted in the optical system near the eye-piece facilitates this observation.

Annealing schedules depend firstly on the hardness of the glass, which determines the high temperature given in the table below, and secondly on the thickness or complexity of the apparatus which determines the rate of cooling. Typical annealing schedules are given below. The apparatus should be heated so that the whole of it is uniformly at the high temperature for a short time. It is then cooled through about 100° C. at a rate not exceeding $20/t^2$ ° C. per minute for soft glasses, where t is the wall thickness in millimetres. For hard glasses a cooling rate of $100/t^2$ ° C. per minute should not be exceeded, the cooling being as uniform a rate as possible. The rate of cooling down to room temperature after this 100° C. drop can then be rapid, as no strain is introduced at this stage. It is best to continue to cool comparatively slowly, however, since the glass may crack, even though no permanent strain can be introduced.

Small vacuum vessels are usually made of soda glass or lead glass. Larger vessels are best made of Pyrex, Monax or Hysil glass. Soda glass is worked in an ordinary gas-air flame at about 900° C., whereas Pyrex glass needs an oxygen coal-gas flame at about 1400° C.

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Type	Name and reference number	High annealing temperature °C.	Thermal expansion coefficient $\times 10^{-7}$	Specific gravity
Soda-lime	Wembley X8	520	90	2.5
	Chance GW1	520	87	2.5
	Plate glass	530	85	2.5
Lead	Wembley L1	430	93	3.0
	Chance GW2	420	86	3.0
	Lead crystal	400	94	3.2
Hard	Hysil GH1	590	33	2.24
	Pyrex	590	33	2.24
	Wembley W1	570	40	2.32
	Intasil GS1	580	39	2.30
	Chance GS3	480	48	2.25
	Chance GS4	600	45	2.32

If two glasses of different coefficients of linear expansion with temperature are fused together the joint will crack on cooling. A graded seal is essential to ensure a permanent join, with one, two or more intermediate glasses, the number of intermediates required depending on the difference in the thermal expansions of the two outside glasses. Thus fusing Pyrex to soda glass requires some four intermediate glasses whose expansions increase progressively from that of the glass with the least expansion to that with the greatest expansion. Not only must the coefficients of expansion be carefully graded, they must also be close together in value over a wide range of temperature for neighbouring glasses in the join.

Likewise glass-to-metal seals depend on their relative coefficients of expansion, and also on the necessity that the fused metal must "wet" the glass surface. The commonest metal seals to glass used in vacuum practice are :

To soft glasses :

Platinum.

Copper-clad or Dumet wires, not more than 1 mm. diameter.

"Feathered" copper edges, as in Housekeeper joint.

26% or 28% Chrome-iron alloys.

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To hard glasses :

Tungsten (to Pyrex, and Chance W1, GS1).

Molybdenum (to Pyrex and Hysil, but better to special glasses like GS4, HH, C11).

Nickel-cobalt-iron alloy such as Kovar, Fernico, Darwin F, etc., to special glasses containing soda, lime and magnesia, like Chance GS3, C40, etc.

Thin borated copper, as in the Housekeeper joint.

If a fused joint between metal and glass is impossible, the alternative is a greased or waxed ground glass cone or flange joint. Hard wax, like picein or "W"-wax will readily effect a join between a glass tube inserted inside a metal tube of slightly greater diameter.

Some physical constants for glass have been given in the above table. Others can only be given approximately, because they vary widely according to the manufacturer of the glass and the method of test.¹

Average breaking stress : 3 to 7 kg./mm.²

Stress for 1% risk of breakage : 1 to 2 kg./mm.²

Specific heat : 0·18 to 0·23 cal./gm./°C.

Thermal conductivity : 1·7 to $3\cdot0 \times 10^{-3}$ cal.cm.⁻¹sec.⁻¹°C.⁻¹

Electrical resistivity : 10^8 to 10^{16} ohm-cm.

These figures are quoted for normal temperatures. Rise of temperature has little effect on tensile strength, or specific heat, but thermal and electrical conductivity both rise rapidly with temperature.

The specific heat of glass can be calculated from the formula

$$S = \frac{1}{100} \sum P S^1 \text{ where } P \text{ is the percentage of the constituent and } S^1 \text{ its specific heat (Winkelmann).}$$

Values of S^1 for various constituents of glasses are : SiO₂ 0·19 ; B₂O₃ 0·23 ; ZnO 0·12 ; PbO 0·051 ; MgO 0·2439 ; Al₂O₃ 0·2074 ; As₂O₃ 0·1276 ; BaO 0·068 ; Na₂O 0·2674 ; K₂O 0·186 ; Li₂O 0·55 ; CaO 0·19 ; P₂O₅ 0·19 ; Mn₂O₃ 0·17.

The viscosity of glass varies widely with temperature. At the melting or founding temperature the viscosity is between 1300 and

¹ Viscosity, thermal expansion and electrical resistivity are composition sensitive, but mechanical strength and thermal conductivity do not change greatly from glass to glass (see R. W. Douglas, *Jour. Sc. Inst.*, 22, 81, 1945).

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1400 poise for soda glass, but this figure varies widely with the glass composition. At annealing temperatures, which can be defined as at specific viscosities, values of 10^{13} to 10^{14} poise prevail.

Glass fluoresces under the action of radiation, or electron or ion bombardment. Such fluorescence is very marked in devitrified glass.

The transmission of visible light through glass varies slightly with the type. Generally speaking, ultra-violet light at wave-lengths below 3000 Å.u.s is virtually cut-off by a glass block 1 cm. thick, though in the case of some high-silica optical glasses the transmission at this wave-length may be as much as 50%.

When glass is melted in vacuum at 1400° C. gases such as water-vapour, oxygen, carbon dioxide, and sometimes sulphur dioxide, are given off.

According to R. W. Douglas¹ the quantity of gas evolved from glass when heated in vacuum (e.g. in roasting a glass vacuum tube on pumps) is a function of the glass composition and the condition of storage since manufacture. Increase of alkali content increases gas to be removed. Best stored in warm, dry place. Washing, in soap and water, before use, alleviates deleterious effects of storage, but rarely eliminates it if a pattern is produced by weathering. Lime-soda glass when heated to 300° C. can reach stage when no further evolution of gas occurs, whilst at 400° C. gas continues to be evolved after some hours. Latter effect is said to be due to either diffusion of air through the hot glass or due to gas evolved from chemical changes in glass. It is extremely unlikely that glass exhibits any porosity to air, however, even at high temperature.

Alpert and Buritz² show, nevertheless, that an ultimate limitation on the achievement of very low pressures in glass systems is caused by the diffusion of atmospheric helium through the walls.

Gold. Evaporated or sputtered films of gold form a useful light filter of readily controllable density and with selective transmission in the green.

Gold is useful to a small extent in place of silver to form photocathodes. Useful as a metal film, not easily broken down, or aggregated, by temperature increases.

¹ R. W. Douglas, *ibid.*

² Alpert and Buritz, *Jour. Appld. Physics*, **25**, 202, 1954.

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A film of gold 5×10^{-6} cm. thick gives the following transmissions :

Wave-length of incident light in A.U.S	Transmission %
4000	4.5
5000	10
6000	4
7000	2

A colloidal suspension of gold in oil can be spread in a thin film on a refractory material. The oil is baked off at about 300° C., leaving the gold film.

Greases : see **Cements.**

Iron. Drawn steel is used in mercury diffusion pump manufacture as it is unattacked by mercury.

Pure Svea iron is used in electron tubes for valve anodes.

Iron-chrome is a useful alloy which seals readily to ordinary glasses : used for lead-in connexions through glass seals, and for water-cooled anodes of transmitting valves up to 250 W, employing a fused seal between iron-chrome anode tube and glass tube holding pinch.

Kovar. Like Fernico, though has not identical properties. Composition : 53.7% Fe, 29% Ni, 17% Co, 0.3% Mn. Melts at 1450° C. After annealing, coefficient of thermal expansion is 5.7 to $6.2 \times 10^{-6}/^{\circ}\text{C}$. in range 30° to 500° C.

Before sealing to glass, or brazing or soldering to other metals, the Kovar should be de-greased, rinsed in alcohol, dried and heated to 900° C. in a hydrogen furnace to out-gas and anneal it. It is then best copper-plated where the join is to be made. It may be brazed to steel or nickel using pure silver or copper in an hydrogen furnace. It should never be heated above 1100° C.

Lead. As an alternative to a rubber gasket between a flange and cover plate a lead gasket can be used. If a ring of lead wire over $\frac{1}{8}$ " diameter is placed between a steel flange and flat plate, and pressure applied by screw clamps, a vacuum-tight join can be obtained.

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Lead is the only metal which satisfactorily seals to silica. It is used for the thick lead-in wires to electrodes of transmitting valves.

Magnesium—used as a getter material.

Magnesium evaporated on to glass with a top layer of evaporated aluminium gives a hard, highly reflecting front surface mirror superior in its adhesion properties to a pure aluminium film.

Usually obtained in form of magnesium ribbon. Oxidizes rapidly in air: must be kept in a vacuum desiccator or alcohol if to be used in vacuum.

Mercury. Extensively used in manometers, diffusion pumps, cut-offs, switches, discharge lamps.

Temp.	- 78° C.	0° C.	20° C.	60° C.	100° C.
Satn. vapour pressure in mm. Hg	3×10^{-9}	2×10^{-4}	$1 \cdot 3 \times 10^{-3}$	3×10^{-2}	$2 \cdot 8 \times 10^{-1}$

Mercury-vapour discharges are employed in the mercury-vapour rectifier and thyratron gas-filled relay triode valves. The anode currents at anode voltages above the striking potential of mercury are much in excess of those in hard vacuum valves because the positive mercury ions neutralize the electron space-charge.

Small rectifiers with a 12-watt cathode give currents up to 250 mA. Large mercury-vapour diodes in an envelope of steel continuously pumped handle up to 75 amps. at 15,000 inverse peak volts.

Mercury lamps consisting of an arc in mercury vapour are useful for illumination purposes. In a silica tube, a mercury arc produces strong ultra-violet radiation. Both singly charged ions Hg^+ and doubly charged ions Hg^{++} (atom less two electrons) can be produced in mercury vapour depending on potential applied.

A mercury switch consisting of a small tube partially filled with mercury in which contact between two sealed-in electrodes depends on angle of tilt of the tube is very convenient in many automatically operated circuits, e.g. such a mercury switch can be closed by the jet of water issuing from a water-cooled diffusion pump; if the water supply accidentally fails then the switch tilts and electric heater supply to pump is cut off.

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Surface tension of mercury = 475 dynes/cm. at 20° C. in vacuum.
= 500–400 dynes/cm. in air.

Mercury readily combines with most metals at room temperature to form amalgams. Iron, cobalt, nickel, platinum, rhodium, molybdenum and tungsten are not readily affected.

Mercury can be cleaned by following methods :

1. Remove large particles by squeezing through chamois leather.
2. Remove metallic impurities by sprinkling down a long column of nitric acid (3 parts water to 1 part acid).
3. Much dirt and grease can be released to form a readily removed scum on surface by bubbling air through the mercury.
4. Distil mercury in vacuum.

The last process should always be employed for mercury to be used in pumps and gauges.

Mercury vapour can cause poisoning of the blood-stream if inhaled.

Mica. An insulator much used for supporting electrodes in vacuum tubes, and as a dielectric in condensers.

Specific resistance = 10^{16} ohm-cm.

Dielectric constant = 5.7 to 7.0

Dielectric strength = 80–200 kV./mm.

Thermal conductivity = 1.8×10^{-3} cal.cm. $^{-1}$ sec. $^{-1}$ °C. $^{-1}$

Mica occurs naturally in the forms of muscovite and biotite. Muscovite-mica which is practically colourless can be obtained. Biotite-mica is much discoloured. Mica can be split along the cleavage planes into very thin sheets. Laminæ 0.0005" thick can be obtained.

Before use in a vacuum tube should be baked to 200° C. to free it of water vapour. Mica surfaces can be regarded normally as being saturated with gas ; mica contains 18% combined water.

Electric leaks across mica, due to volatilized metal films caused during out-gassing of electrodes in a vacuum tube, are reduced by spraying alumina insulation on the mica so that any deposited metal will not form a continuous conducting surface.

If baked above 800° C. mica loses its normal form, cleavage planes separate and colour appearance of silver-grey is assumed.

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Evaporated silver films on either side of a mica sheet are used for producing accurate low-capacity condensers ($< 0.001 \mu\text{F}$). A resistance of desired value can be obtained by evaporating a layer of gold or platinum of appropriate thickness on to mica.

Metal films evaporated on to mica do not adhere strongly ; rubbing with a cloth will readily remove them.

A mica sheet 0.001" thick transmits approximately 90% of normally incident white light. Wave-lengths below 2600 A.U.S are cut off.

Molybdenum. Used particularly for transmitting valve anodes, X-ray anti-cathodes, and grid wires in receiver valves. Wires down to 0.025 mm. diameter can be drawn. Seals to borosilicate glasses in wire diameters up to 8 mm.

Electron emission in mA./cm.² :

At 1000° C.	1630° C.	2230° C.
10^{-9}	8.3×10^{-1}	800

Molybdenum is more flexible and readily shaped than tungsten. Can be spot-welded to iron and nickel. A tungsten to molybdenum, or molybdenum to molybdenum electric weld is practically impossible. Some adhesion can be obtained if a thin grease layer is spread over the metals.

Molybdenum heated to 1000° C. readily absorbs oxygen.

Cleaning of molybdenum preparatory to its use *in vacuo*, or for making glass-molybdenum seals, can be done by heating in contact with sodium nitrite at 800° C. approx.

A tungsten-molybdenum wire consisting of 51% Mo is a standard valve filament or cathode heater wire. Melting-point = 2900° C. Has greater strength than molybdenum, and more readily shaped than tungsten.

Nichrome : *see Nickel.*

Nickel. Most used metal for vacuum-tube electrode construction since it is readily cleaned and out-gassed, has a high melting-point, is comparatively difficult to corrode, and is readily welded to most metals.

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Seals to glass, but will not produce a vacuum-tight seal.

Nickel is ferro-magnetic : max. permeability = 700 gauss/oersted
coercivity = 6 oersted

Magnetic permeability very small above 300° C.

Nichrome is an alloy : 64% Ni, 11% Cr, 25% Fe.

Melts at 1350° C. The wire is much used in electric heaters, ovens and furnaces.

Resistivity at 20° C. = 110×10^{-6} ohm-cm.

Temp. coeff. of resistance = 1.7×10^{-4} per ° C.

Invar is an alloy of 63% Fe, 36% Ni, 0.5% Mn, used whenever very small coefficient of expansion desirable.

Coeff. of linear expansion = 4×10^{-7} per ° C.; increases considerably above 100° C.

Melting-point = 1425° C.

Palladium. A noble metal of the platinum group. Chiefly used as a getter and source of supply of hydrogen since palladium dissolves hydrogen to form palladium hydride. At N.T.P. 6 mg. hydrogen are absorbed by 100 grammes of palladium.

At -190° C. absorbing power greatly increased.

If required for gas absorption, the metal must be used in form of palladium black prepared by dissolving in aqua regia, then baking and adding sodium carbonate to free compound of acid. Then warm solution adding acetic acid, and afterwards add a warm concentrated solution of sodium formate. The palladium will precipitate in form of palladium black which has to be washed and dried. It oxidizes if heated in air, and is soluble in nitric acid.

If temperature raised beyond 300° C. hydrogen is re-evolved.¹

Phosphorus. A much used getter in early history of vacuum practice. Campbell introduced phosphorus into vacuum tube via a side-tube. Red and white forms of phosphorus exist. A filament can be coated with red phosphorus by dipping in a suspension of phosphorus powder in alcohol plus cementing sodium silicate. Heated in vacuum the filament readily absorbs the commoner gases.

The use of phosphorus as a getter has passed out of favour because it acts chemically on the thermionic and photo-cathodes commonly used in electron tubes.

¹ Dunoyer, *Vacuum Practice* (G. Bell and Sons Ltd., 1926).

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Temp. in ° C.	0	30	80	100
Vapour pressure in mm. Hg of white phosphorus .	7×10^{-3}	7.1×10^{-2}	8.23×10^{-1}	3.6

Phosphorus Pentoxide is the most used drying agent. The powder readily absorbs water vapour. See "drying agents", Chapter Four. Vapour pressure of phosphoric acid formed by saturating pentoxide with water = 5×10^{-4} mm. Hg approx.

Platinum. A noble metal, inert to all acids but aqua regia. Employed whenever long use without corrosion desirable. Seals readily to common glasses to give vacuum-tight join. Often sandwiched between copper and glass to form seal. Sputters readily, but is difficult to evaporate *in vacuo*. (See Chapter Five.)

Platinum refractory crucibles for chemical actions obtainable; used as a wire in standard flame-tests in chemical analysis.

Above 700° C. platinum foil will readily allow passage of hydrogen.

Very ductile; wire of diameter 0.02 mm. obtainable. Wollaston sheathed platinum wire in silver tube and drew fine wire through dies: the silver etched from platinum core leaves a wire as small in diameter as 5×10^{-4} mm.

A layer of platinum sputtered on to glass forms a useful mirror. Baking a Pyrex glass at 10° C. below the melting-point on which a platinum film has been formed will "sinter" the platinum into the glass. No amount of scratching or rubbing will remove such a film. Used in manufacture of high-grade optical graticules, and for sputtered electric resistors.

Reflectivity of a platinum film on glass:

λ in Å.U.S	Percentage of normally incident light reflected
3700	40
3850	45
4200	52
5600	50
6000	64
8000	70

¹ International Critical Tables, 1929.

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Potassium. An alkali metal which reacts strongly with air at normal temperatures. Used in manufacture of special types of photo-cells. A potassium oxide photo-cell has a high green and blue sensitivity, but is insensitive at $\lambda > 8000$ A.U.S. Potassium hydride cells are selectively blue sensitive with a maximum at 4400 A.U.S. Otherwise overall white light sensitivities are less than for caesium cell.

A potassium trap can be used in place of a liquid air trap in a mercury-diffusion pump system, but it is by no means convenient. The metal is distilled and condensed in a thin film on the inside of the trap. Absorbs up to its own weight of mercury, but does not absorb other condensable vapours.

(See Hughes and Poindexter, *Phil. Mag.*, **50**, 423, 1925, also Hunten, Woonton and Longhurst, *Rev. Sc. Inst.*, **18**, 842, 1947.)

Quartz : see Silica.

Rhodium. A metal very similar to platinum but cannot be readily drawn into wire form. Chief use in electro-plating: forms best corrosion-free surface. Recently has come to fore in form of a rhodium film evaporated on to glass. (See Chapter Five.) Some 80% of incident white light is reflected, and front-surface mirror so formed is extremely hard and adhesive, so that it can be used for naval and aircraft optical instruments which are subject to severe weathering. Also the film is practically neutral, i.e. it reflects all parts of visible spectrum uniformly and, moreover, a transparent thin film transmits to a uniform extent radiation in region 4000 to 8000 A.U.S

λ in A.U.S	Percentage of normally incident light reflected
3,000	73
4,000	79
6,000	81
8,000	83
10,000	85

Rubber is used in the forms of rubber tubing and rubber gaskets. A low sulphur content is essential. Before use it must be thoroughly

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cleaned ; boiling in caustic soda solution, then distilled water and drying in an air-blast is a satisfactory treatment.

Rubber tends to give off much hydrogen. The vapour pressure of cleansed, de-gassed rubber is of the order of 10^{-4} mm. Hg. For use as a gasket a semi-hard rubber (India rubber) is best. The surface area should be considered in cutting the gasket : the lower it is the greater will be the pressure per unit area squeezing the rubber to give a gas-tight seal. A gasket between a flat plate and bell-jar with ground flange can be relied on to enable a pressure down to 10^{-5} mm. Hg to be obtained.

Rubber tubing should be confined to the fore-pressure parts of a vacuum system.

Thermal conductivity of pure rubber

$$= 3.1 \times 10^{-4} \text{ cal.cm.}^{-1}\text{sec.}^{-1}\text{C.}^{-1}$$

Resistivity = 10^9 ohm-cm. approx.

Young's modulus = 5×10^9 dynes/cm.²

Rigidity modulus = 1.5×10^7 dynes/cm.²

Density (pure India rubber) = 0.92.

SYNTHETIC RUBBERS. The most satisfactory artificial rubber for vacuum purposes is *neoprene*, since it has a strength and toughness comparable with real rubber. It is also resistant to the action of oils and other organic solvents, and withstands temperature increases in much the same way as the natural product. It is costly because control and manipulation in the plastic state are relatively complicated.

Thiokol has good resistance to the action of oils, but has not the strength and elasticity of natural rubber, and is affected more by changes of temperature. Sulphur appears in the molecule. Its only real advantage over neoprene is that it has greater swelling resistance.

The *Bunas* : two varieties Buna-S and Buna-N exist, the S variety being the more plastic. The physical properties of the Bunas are distinctly inferior to those of natural rubber.

Cloud and Philp,¹ in making tests on rubber O-ring gaskets to seal off a vacuum system, show that rubber evolves large amounts of gas compared with a lead gasket. The apparent vapour pressure recorded was 10^{-3} mm. Hg at 20°C. , which decreased to 10^{-4} mm.

¹ R. W. Cloud and S. F. Philp, *Rev. Sc. Inst.*, **21**, 731, 1950.

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Hg¹ at the temperature of liquid air (-187° C.). Continuous pumping for several days produced no improvement, nor did the use of metal-to-metal joints including a rubber gasket, with a minimum exposure of the rubber to the vacuum system. An equilibrium pressure of 10^{-5} mm. Hg was attained after pumping a rubber-sealed vessel of $\frac{1}{2}$ litre volume with an oil diffusion pump having a speed of 25 litres/sec. at the chamber. This reading was obtained with an ionization gauge, where a trap cooled with solid CO₂ was between the chamber and the gauge. On using a liquid air trap, the pressure fell to 5×10^{-6} mm.

It must be remembered that an ionization gauge gives a reading depending on the ionization probability of the atmosphere in which the gauge is operating. The figures quoted are hence only approximate. They are nonetheless significant.

Rubidium. An alkali-earth metal similar to caesium. Chief application is in rubidium-oxide photo-cell giving a spectral sensitivity approximating to that of the human eye, with a maximum in the green.

Silica. Amorphous silicon dioxide; the crystalline variety is quartz: a mineral. Fused silica used for transmitting valve envelopes; ultra-violet photo-cell tubes. Fused quartz fibres are much used for measuring instrument suspensions. Crystalline quartz slabs exhibit the piezo-electric effect enabling them to control rigidly the frequency of an oscillatory circuit.

Fused silica is inert to all liquids except hydrofluoric acid.

Density of fused silica = 2.1-2.2.

Coefficient of heat expansion = 0.42×10^{-6} per $^{\circ}$ C.

Young's modulus = 5.2×10^{11} dynes/cm.² increasing up to 11.0×10^{11} for thin fibres down to 3×10^{-3} mm. in diameter.

Rigidity modulus = 3.0×10^{11} dynes/cm.² again increasing up to 6.5×10^{11} for thin fibres.

Silica is harder than glass and most metals. Melting point = 1700° C. Silica has to be worked in an oxy-coal-gas or oxy-acetylene flame. Re-crystallization tends to occur at 1200° C. Lead is the only metal which satisfactorily fuses to silica.

¹ This does not imply that the true vapour pressure of rubber is so high at such low temperatures. Rather it implies that rubber, at 20° C., is giving off gases not condensed in the liquid air trap.

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Thermal conductivity = 24×10^{-4} cal.cm. $^{-1}$ sec. $^{-1}$ C. $^{-1}$

Resistivity = 5×10^{18} ohm-cm. at 20° C., and
at 600° C. = 6×10^7 ohm-cm.

Dielectric constant = 4.5 for crystal quartz.

= 3.7 for silica or fused quartz.

Refractive index = 1.45843 at $\lambda = 5893$ A.U.S and 18° C.

Quartz is much used in optics when a high ultra-violet transmission is required. Useful transmission down to $\lambda = 2000$ A.U.S and up to 35,000 A.U.S in infra-red is obtained.

A thin layer of silica evaporated on to an optically-worked glass surface *in vacuo* can be used to protect the glass against the chemical action of weathering (see Chapter Five).

Silicon monoxide evaporates *in vacuo* at lower temperature than dioxide. Prepared by heating silica with silicon at pressure of $< 10^{-4}$ mm. Hg, when



Silica-Gel is a useful drying agent, though not so effective as phosphorus pentoxide. It has one advantage over pentoxide in that it can be restored to activity by baking, and so can be used many times.

Silver. Readily sputtered and evaporated in vacuum to form metallic films on a support.

At λ in A.U.S	Percentage of normally incident light reflected
3,000	≤ 30
4,000	86
6,000	94
8,000	94
12,000	94

Figures are for freshly evaporated pure silver film on optical flat.

Best reflector for white light.

Evaporated silver films are not strongly adherent.

Silver can be readily deposited on glass from chemical solution by Rochelle Salt and Brashear Process. Silver is the best conductor

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of heat and electricity, and is, for these reasons, very difficult to weld electrically to other metals. For silver chloride, see **Cements**.

Sodium. An alkali metal, usually prepared for vacuum technique (e.g. for a sodium photo-cell) by reacting sodium chloride with calcium.

Sodium lamps employ a discharge through sodium vapour and emit a yellow light. Sodium vapour also fluoresces.

Sodium is conveniently soluble in liquid ammonia.

The sodium photo-cell is active to a narrow band of wavelengths from 2000 to 5000 A.U.S with a pronounced maximum at 3300 A.U.S.

The emission spectrum of sodium—yellow light :

two pronounced lines at $\lambda = 5889\cdot965$ A.U.S (D_2)
and $\lambda = 5895\cdot932$ A.U.S (D_1).

R. W. Douglas¹ describes a special sodium vapour resistant glass which is so soft and quick-setting that it can be used as a "flashed" layer on the inside surface of another glass tube.

Solders. *Soft solder* is half tin and half lead, melting at 188° C. Often supplied in form of a resin-cored wire. Best flux is zinc chloride with ammonium chloride.

Wood's metal is a low-temperature solder consisting of 50% Bi, 12·5% Cd, 25% Pb, and 12·5% Sn which melts at 60° C. Useful whenever join required which must not be excessively heated.

Silver solder gives most robust join. Consists of 45% Ag, 30% Cu and 25% zinc. Melts at 720° C., and used with borax and boracic acid flux.

Brazing compound, consisting of 54% copper and 46% zinc, melts at 875° C.

A glass-tombac join can be made using soft solder, or Wood's metal, and borax flux, if the glass is previously coated with silver or platinum.

Steels. Iron mixed with a small percentage of carbon. Mild steel is usually employed in construction of large vacuum systems. The properties of steel are much altered by alloying with other metals.

¹ R. W. Douglas, *Jour. Sc. Inst.*, 22, 81, 1945.

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Steel containing chromium, nickel-chromium, nickel-copper, vanadium, manganese and silicon are common. Less-used steels having particular properties are aluminium, arsenic, bismuth, cerium, antimony, tantalum and zirconium steels. For a full account of the many types ref. *Int. Crit. Tables*, vol. II, pp. 483-532, 1927.

Following are average constants : Density 7.7 to 7.9 gm./cm.³

Steel (1% C) Young's modulus = 20.9×10^{11} dynes/sq. cm.

Rigidity modulus = 8.12×10^{11} dynes/sq. cm.

Breaking stress in dynes/sq. cm. :

for cast steel = 2.3 to 7.0×10^9 dynes/sq. cm.

mild steel (0.2% C) = 4.3 to 4.9×10^9 " "

spring steel (high % C) = 7.0 to 7.7×10^9 " "

5% tungsten steel = 6.2×10^9 " "

12% CrNi steel = 14.0×10^9 " "

Thermal conductivity (1% C) steel = $0.115 \text{ cal.cm.}^{-1}\text{sec.}^{-1}\text{C.}^{-1}$

Linear expansion coeff. " = 10.5 to 11.6×10^{-6}

Specific resistance " = 19.9×10^{-6} ohm-cm.

	Magnetic coercivity in oersted
For mild steel (0.2% C)	0.4
Cobalt steel	200
Tungsten steel	80
Chromium steel	56
Molybdenum steel	85
Wrought iron	0.8
Cast iron	4.6

Magnetic induction for mild steel at maximum magnetizing field = 18,190 gauss. With a magnetizing field = 100 oersted, the induction = 17,700 gauss. Maximum permeability = 8350 C.G.S. units.

Strontium. An alkali-earth metal similar to barium. The oxides of strontium and barium are mixed to form the standard thermionic cathode coating.

Strontium oxide has vapour pressure of 10^{-6} mm. Hg at 1400° C.

Barium oxide " " " " " 10^{-6} " " " 1150° C.

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Sulphur. Very little used in vacuum practice. Is a very good electrical insulator : resistivity at 70° C. = 7×10^{15} ohm-cm.
,, at 20° C. = 10^{17} ohm-cm.

The presence of sulphur is ruinous to most vacuum operations : removed as an impurity (e.g. from rubber) by dissolving in caustic soda solution.

Tantalum. A highly refractory metal used for transmitter valve anodes where a large heat dissipation occurs. Getters hydrogen, oxygen and nitrogen at temperatures around 600° C., these gases being re-evolved above 800° C. Offers great resistance to corrosion and chemical attack, withstanding all acids except hydrofluoric and fuming sulphuric at normal temperatures, but is attacked by strong alkalis.

Electron emission in mA./sq. cm. at 1000° C. is 10^{-5} , at 1250° is 4.7×10^{-3} and at 1750° it is 19.5. The work function for tantalum is less than that for tungsten, but greater than that for columbium.

The mechanical strength of tantalum is comparable with that for steel, both in the fine wire and thin sheet forms. The yield and breaking points are practically identical for the two metals. Machining is carried out with ordinary machine tools, using carbon tetrachloride for cooling.

Welding of tantalum to itself and a number of other metals is possible by electric spot-welding and special arc-welding methods.

Tantalum carbide is an extremely hard substance which melts at approximately 4000° C. It is used with the carbides of other metals such as tungsten, for cutting tools, making wear-resistant parts, and for dies in wire-drawing apparatus.

Thorium. Admixture of thorium with tungsten gives a thoriated tungsten filament which emits electrons more efficiently when heated than pure tungsten. A filament containing 2% thorium oxide (ThO_2) is heated for 2 minutes at 2500° C. *in vacuo*, and then maintained at 2000° C. approx. to cause "activation". During this "activation" the emission rises from 1 mA./sq. cm. at the operating temperature 1730° C. to 2.75 amps./sq. cm. The thorium diffuses to the surface of the tungsten and is the actual emitter.

Tin. Molten tin can be used to join a glass to metal tube providing

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they overlap for about 20 mm. of their length. If the glass is first sputtered with a thin layer of platinum, the join is facilitated.

Titanium. G. Hass¹ gives data on the oxidation of evaporated titanium films. Titanium metal may be evaporated *in vacuo* from a directly electrically heated tungsten strip at 2000° C., currents of the order of 200 A. being demanded. Pure titanium films are then produced if the pressure is 10⁻⁵ mm. Hg or less. The oxidation of such pure titanium films by heating in air at 400° to 450° C. results in titanium dioxide films with rutile structure and having high refractive index. Such films are valuable in multi-layer thin film combinations on glass in the manufacture of interference filters in optics, also in producing enhanced reflecting layers (e.g. on lithium fluoride on glass) of low light absorption, useful in optical beam-splitter applications. Anodically oxidized titanium films might also well be valuable in providing a hard, transparent layer on aluminized mirrors. The thickness of the titanium dioxide film produced can be precisely controlled by the baking time in conjunction with the time of deposition of the titanium. Adhesion to glass is very great. Slow evaporation of titanium at low temperature in poor vacuum results, after baking in air, in TiO-TiN films with cubic structure and of little value in optics.

Tombac: *see Brass.*

Tungsten. The metal with the highest melting-point. Used for lamp and valve filaments and electric heaters. Has needle-like crystal structure making bending difficult without splitting, but has greater tensile strength than any other metal. Available in thin sheet form, useful for strip heaters. The German product is much more malleable than the British.

When heated acts as a getter to residual oxygen and carbon monoxide in an evacuated vessel.

Seals readily to Pyrex and Monax glasses. Vacuum-tight Pyrex-tungsten seals around tungsten wire up to 2 mm. in diameter are much used. To make such a seal the tungsten must be first cleaned by rubbing when hot with sodium nitrite. Alternatively, tungsten can easily be cleaned by de-plating. It is made the anode in a beaker containing potassium hydroxide, with some other electrode

¹ G. Hass, *Vacuum*, 2, 331, 1952.

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as cathode. A 6-V. accumulator is a satisfactory electric supply. Tungsten surface oxidizes readily at room temperature.

Electron emission in mA./sq. cm. at 830° C. = 1.5×10^{-10}
 at 1630° C. = 2.3×10^{-1}
 at 2230° C. = 298.

Young's modulus = 39×10^{11} dynes/sq. cm.

Rigidity modulus = 15×10^{11} " "

These figures vary since drawn tungsten wire (down to 0.0005" dia.) exhibits greater ductility.

Water

Temp. °C.	Density grm./cm. ³	Viscosity poise	Surface tension dynes/cm.	Vapour pressure mm. Hg	Expansion coefficient	Refractive index at $\lambda = 5893$ A.U.S
-187	—	—	—	—	—	—
-78	—	—	—	6×10^{-4}	—	—
0	0.99984	1793	—	4.58	—	—
4	0.99997	—	74.92	—	—	—
10	0.9997	1309	74.22	9.2	0.00015	0.33374
50	0.9880	550	—	92.5	0.00046	—
100	0.95835	284	—	760	0.00070	—
200	0.8628	—	—	11,700	—	—

Melting point = 0° C. Boiling point = 100° C. at 760 mm. Hg ; or at pressure p , boiling point = $100 + 0.0367 (p - 760)$.

Thermal conductivity = 1.4×10^{-3} cal.cm.⁻¹ sec.⁻¹ °C.⁻¹

Electrical conductivity = 10^{-6} ohms⁻¹ cm.⁻¹ for distilled water.

Dielectric constant = 81 at 20° C., for distilled water.

Solubility of gases in water :

Gas	Volume of gas at N.T.P. absorbed by 1 c.c. of water at 20° C.
Air	0.019
Ammonia	700
Argon	0.038
Carbon dioxide	0.88
Helium	0.0138
Hydrogen	0.0181
Nitrogen	0.0154
Oxygen	0.031

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Wood's Metal : *see Solders.*

Zinc. Zinc oxychloride : a paste of 60% zinc chloride solution and zinc oxide powder forms a useful cement.

Zirconium. A ductile and malleable metal unless it contains absorbed oxygen and nitrogen. Acts as a getter material : operated at 1400° C. absorbs oxygen, nitrogen, carbon monoxide and carbon dioxide. At 300°–400° C. absorbs hydrogen. Two wires heated in vessel, one to 400° C. and the other to 1400° C., act as fairly complete getter. Can be put on anode of transmitter valve and heated by electron bombardment. Has low secondary emission ; useful on valve grids to prevent grid emission.

4 parts of TaC with 1 part ZrC forms substance with highest melting point = 4000° C.

Powder form zirconium prepared by reacting zirconium chloride with sodium.

A filament of zirconium is prepared by thermal decomposition of zirconium tetra-iodide on a glowing filament of tungsten.

(*See Philips Tech. Rev.*, Dec. 1938 ; "Zirconium", by J. D. Fast.)

VAPOUR PRESSURES OF COMMON LIQUIDS

Liquid	Vapour pressure at 20° C. in mm. Hg
Acetone	180
Alcohol (ethyl)	44.5
,, (methyl)	88.7
Benzene	74.6
Bromine	172
Carbon dioxide	42.9
Carbon tetrachloride	91
Ethyl ether	440

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